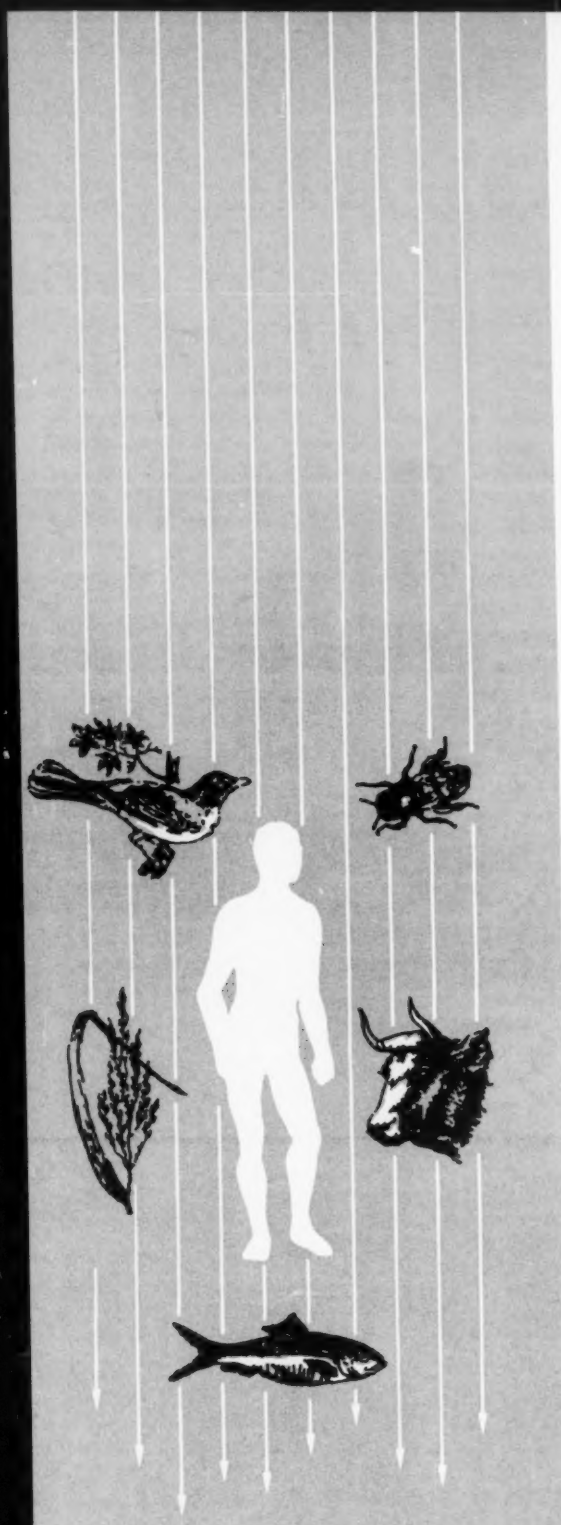

PESTICIDES MONITORING JOURNAL

JUNE 1978

VOLUME 12 NUMBER 1

PEMJAA (12) 1-46 (1978)

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SOIL

DDT Moratorium in Arizona—Agricultural Residues After Seven Years [1]

George W. Ware, Betty J. Estesen, Norman A. Buck, and William P. Cahill

ABSTRACT

The moratorium on agricultural use of DDT in Arizona that began in January 1969 proved very effective during the first 7 years of enforcement. Residues on green alfalfa declined significantly to a probable inherent level of 0.02 ppm wet weight. Soil residues of ΣDDT-related degradation products declined significantly, averaging 23 percent; residues in desert soils declined 60 percent. The ΣDDT half-life in irrigated soils was about 7 years; it decreased to 2.5 years in nonirrigated soils.

Introduction

The moratorium on agricultural use of DDT in Arizona began in January 1969 (*2, 4, 5*). This is the fourth and probably last report on the status of DDT residues and ΣDDT-related degradation products, after 18 years of unrestricted use and 4 years of restricted use in Arizona.

Analytical Methods

Alfalfa and soil samples were collected as described in previous reports (*2, 4, 5*) from the three major irrigated areas in Arizona: Salt River Valley, which surrounds Phoenix; Pinal County; and the Yuma mesa and valley in Yuma County. Desert soil samples, but only the top 0.25 inch, adjacent to these areas were also collected. In addition an earlier study (*3*) was continued to provide reference standards and continuity for the seven-year period (Table 1). The sampling sites are located on a 60-mile Maricopa County east-west transect along Baseline Road, much of which is now residential.

Alfalfa and soil samples were extracted and cleaned by procedures previously described (*2–5*).

TABLE 1. *ΣDDT residues in green alfalfa, Baseline Rd., Maricopa Co., Arizona, 1967–75*

	ΣDDT Residues, ppm						
Sample	1967 Aug.	1968 Sept.	1969 Sept.	1970 Sept.	1971 Sept.	1972 Sept.	1975 Oct.
2	—	0.220	0.038	0.050	0.020	0.023•	0.009•
3	0.283	—	0.027	0.030	—	0.025•	0.007•
4	0.170	0.120	0.038	0.037	0.031	0.022	0.016•
5	—	0.060	0.020	0.024	0.011	0.029•	0.009•
6	0.277	—	0.035	0.022	—	0.008•	—
8	0.794	—	—	0.027	0.038	0.013•	0.023
9	—	0.076	0.034	0.042	0.020	0.029•	0.027
10	0.350	0.092	0.054	0.162	0.027	0.031	0.022•
11	0.453	0.580	0.064	0.047	0.085	0.056	0.027•
12	0.299	0.077	0.025	0.038	—	0.023•	0.014•
13	0.606	—	—	0.021	0.027	—	0.008•
Means	0.404d	0.175c	0.037b	0.045b	0.032b	0.026b	0.016a

NOTE: — = no sample analyzed.
• = substitute adjacent fields.
Means with same letter are not significantly different at the 0.05 level.

Samples were analyzed by electron-capture gas-liquid chromatography (EC-GLC). Recovery standards and analytical reagent blanks were also extracted and cleaned each day. Recoveries were consistently above 90 percent; however, the data presented have not been corrected. The minimum sensitivity of the method was arbitrarily set at 0.02 ng for *p, p′-* and *o, p′*-DDT, DDE, and TDE. Standard curves extended from 0.03 ng to 0.10 ng. The sensitivities were 0.001 ppm for alfalfa and 0.003 ppm for soil. Results are based on a minimum sample size and 6 μl extract injected into the chromatograph.

Analytical EG-GLC confirmatory tests were conducted randomly using a double-length GLC column at the same temperatures as those used in the previous study (*2*). Because of low levels of ΣDDT and interfering peaks of toxaphene which may have drifted from nearby cottonfields, all alfalfa extracts were dehydrohalogenated after cleanup on Florisil and residues were measured only as *o,p′-* and *p,p′*-DDE as described by Cahill et al. (*2*); results were combined when measurable levels of *o,p′*-DDE were found.

[1] Department of Entomology, The University of Arizona, Tucson, AZ 85721. This paper submitted to Regional Project W-45, "Residues of Pesticides and Related Chemicals in the Agricultural Environment—Their Nature, Distribution, Persistence, and Toxicological Implications." University of Arizona Agricultural Experiment Station Journal Series No. 2759.

Results and Discussion

Residues observed in alfalfa and soil samples during the past 7 years are presented in Tables 1–3 as ΣDDT. The Student-Newman Keul's test was used to analyze differences among residue means for the various sampling dates. Comparisons were made on least-square means in the soil samples (Table 3) because there were too few samples. Residues on alfalfa from all four areas shown in Tables 1 and 2 appear to have leveled off at about 0.02 ppm. September values for Yuma County alfalfa were consistently high from 1969 through 1972. However, these values were well below 0.02 ppm in 1975.

Residue levels in alfalfa soils declined from the previous sampling period, September 1972 (Table 3). In the past, yearly examination of these soil residues indicated almost imperceptible changes. After 3 years, however, the residues had declined significantly, an average of 23 percent. Residues in the desert soils declined 60 percent. This suggests that the ΣDDT half-life in the irrigated soils of Arizona is about 7 years, and decreases to about 2½ years in the desert or nonirrigated soils.

ΣDDT residues now found in the agricultural soils of Arizona are shifting steadily toward higher proportions of DDE. The ratio of DDE:DDT in these soils shifted from 56:44 in 1972 to 62:38 in 1975. In the desert soils, the shift was approximately the same: from 65:35 in 1972 to 71:29 in 1975. These data suggest that ΣDDT residues are declining at a predictable rate, probably both by volatility and conversion to metabolites not measured with the analytical methods used in this study.

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TABLE 2. *ΣDDT residues in green alfalfa during 1969–75 DDT moratorium, Arizona*

Sample	ΣDDT Residues, ppm						
	1969 Jan.	1969 Sept.	1970 Sept.	1971 Sept.	1972 Jan.	1972 Sept.	1975 Oct.
Maricopa County, Arizona							
1	0.087	0.042	0.057	—	—	—	0.019*
2	0.303	0.062	0.050	0.025	—	0.039*	0.037*
3	0.102	0.078	0.093	0.038	—	—	0.011*
4	0.107	0.047	0.076	0.037	—	0.046*	0.017
5	0.049	0.030	0.025	0.007	—	0.011	0.015*
6	0.113	0.064	0.060	0.051	—	0.045*	0.041*
7	0.082	0.034	0.023	—	—	0.055	0.21
8	0.125	0.056	—	—	—	—	—
9	0.085	0.044	0.101	—	—	—	—
10	—	—	0.080	0.059	—	—	—
Means	0.117c	0.051b	0.063b	0.036b	—	0.39b	0.023a
Pinal County, Arizona							
1	0.047	0.042	0.034	0.055	—	0.041*	—
2	0.047	0.031	0.059	0.036	—	—	0.068*
3	0.142	0.187	—	—	—	—	0.006*
4	0.231	0.076	0.071	0.072	—	0.025	—
5	0.092	0.130	0.045	—	—	0.025*	—
6	0.038	0.058	0.045	—	—	—	—
7	0.079	0.118	0.059	0.038	—	0.044	0.023*
8	0.068	0.071	0.031	0.034	—	0.018	0.077*
9	0.054	0.068	0.057	0.060	—	—	0.006*
Means	0.088b	0.086b	0.050a	0.049a	—	0.031a	0.036a
Yuma County, Arizona							
1	0.047	0.373	—	0.120	0.025	0.032	0.016*
2	0.039	0.098	—	—	0.010*	0.017*	0.008*
3	0.049	0.256	0.084	0.270	0.073*	0.040*	0.040
4	0.057	0.093	—	—	0.055*	0.075*	0.025
5	0.057	0.545	0.063	0.340	0.047*	0.290*	0.030*
6	0.044	0.317	—	—	0.035*	0.300*	0.032*
7	0.059	0.241	—	—	0.026*	0.190*	0.034*
8	0.036	0.045	0.034	0.031	0.039*	—	0.005*
9	0.021	0.056	—	—	0.015*	—	—
10	0.046	0.074	0.051	0.050	0.028	0.045	0.006
Means	0.46a	0.210b	0.058a	0.162b	0.035a	0.123b	0.022a

NOTE: — = no samples analyzed.
* = substitute adjacent fields.
Means with same letter are not significantly different at the 0.05 level.

TABLE 3. *ΣDDT residues in soils during 1969–75 DDT moratorium, Arizona*

Field No.	1969 Jan., ppm Residues				1972 Sept., ppm Residues				1975 Oct., ppm Residues			
	DDE	*o,p'*-DDT	*p,p'*-DDT	Total	DDE	*o,p'*-DDT	*p,p'*-DDT	Total	DDE	*o,p'*-DDT	*p,p'*-DDT	Total
Maricopa County												
1	0.35	0.04	0.12	0.54	0.40	0.04	0.11	0.55	0.43	0.10	0.13	0.66
2	0.48	0.17	0.78	1.54	0.98	0.18	0.47	1.63				
3	0.33	0.07	0.16	0.59	1.24	0.13	0.32	1.69	1.0	0.10	0.23	1.33
4	0.49	0.05	0.17	0.74	0.58	0.05	0.23	0.86	0.40	0.03	0.12	0.55
5	0.29	0.05	0.09	0.44	0.17	0.01	0.05	0.23	0.17	0.01	0.02	0.21
6	2.10	0.43	1.10	3.93	2.58	0.28	0.96	3.82	2.24	0.31	0.58	3.13
7	0.84	0.11	0.23	1.22	0.92	0.09	0.29	1.30	0.68	0.06	0.14	0.88
8	2.22	0.38	1.29	4.00	2.37	0.27	1.21	3.85	1.96	0.24	0.98	3.18
9	1.18	0.21	0.91	2.41	1.12	0.17	0.77	2.06	0.83	0.14	0.55	1.51
10	—	—	—	(0.24)	0.31	0.04	0.07	0.42	0.24	0.02	0.06	0.32
Means	0.92	0.17	0.54	1.57a	1.07	0.13	0.45	1.64a	0.883	0.11	0.31	1.31b
Desert												
1	0.08	<0.01	0.03	0.13	0.43	0.07	0.09	0.59	0.04	0.00	0.02	0.06
2	0.24	0.02	0.06	0.35	0.28	0.03	0.58	0.89	0.04	0.01	0.02	0.06
3	0.44	0.04	0.15	0.67	0.18	0.02	0.04	0.24	0.06	0.01	0.02	0.10
4	—	—	—	(2.39)	0.54	0.08	0.06	0.68	0.69	0.10	0.19	0.98
Means	—	—	—	0.89a	0.36	0.05	0.19	0.60a	0.21	0.03	0.06	0.30b
Pinal County												
1	0.64	0.48	2.43	3.77	0.74	0.34	2.64	3.72	0.59	0.24	2.51	3.34
2	0.27	0.15	1.03	1.52	0.41	0.13	0.96	1.50	0.37	0.09	1.03	1.49
3	1.05	0.32	1.38	2.75	1.16	0.16	0.80	2.12	0.64	0.09	0.42	1.16
4	0.99	0.27	1.04	2.30	1.40	0.18	0.74	2.32	1.60	0.21	0.25	2.05
5	0.16	0.02	0.21	0.41	0.25	0.02	0.16	0.43	0.19	0.02	0.10	0.31
6	0.06	0.01	0.07	0.14	0.07	0.01	0.04	0.12	0.04	0.00	0.05	0.08
7	1.09	0.28	1.37	2.74	1.63	0.20	0.80	2.63	1.32	0.16	0.31	1.79
8	0.09	<0.01	0.04	0.14	0.08	0.01	0.02	0.11	0.05	0.00	0.01	0.06
9	0.67	0.09	0.29	1.06	0.74	0.03	0.06	0.83	0.59	0.05	0.08	0.72
10	0.66	0.14	0.36	1.16	1.19	0.15	0.39	1.73	0.91	0.09	0.08	1.08
Means	0.57	0.18	0.82	1.60a	0.69	0.12	0.66	1.55a	0.63	0.10	0.48	1.21b
Desert												
1	0.09	<0.01	0.06	0.16	0.17	0.02	0.12	0.31	0.04	0.00	0.01	0.06
2	0.18	0.01	0.11	0.32	0.21	0.02	0.21	0.44	0.07	0.00	0.02	0.09
3	0.05	0.03	0.10	0.21	0.06	0.01	0.02	0.09	0.04	0.01	0.01	0.06
4	0.09	0.03	0.10	0.25	0.77	0.07	0.09	0.93	0.49	0.05	0.07	0.61
Means	0.10	0.02	0.09	0.24a	0.30	0.03	0.11	0.44a	0.16	0.02	0.03	0.20b
Yuma County												
1	0.10	<0.01	0.07	0.17	0.12	0.02	0.03	0.17	0.06	0.01	0.01	0.08
2	0.24	0.05	0.25	0.54	0.20	0.03	0.07	0.30	0.24	0.03	0.06	0.33
3	0.72	0.16	0.72	1.60	0.79	0.16	0.49	1.44	0.71	0.15	0.29	1.15
4	0.59	0.11	0.47	1.17	0.98	0.15	0.46	1.59	0.84	0.13	0.22	1.18
5	0.48	0.05	0.30	0.83	0.75	0.12	0.34	1.21	0.54	0.10	0.15	0.79
6	0.29	0.16	0.74	1.19	0.48	0.10	0.43	1.01	0.45	0.08	0.29	0.81
7	1.29	0.07	0.37	1.73	1.11	0.09	0.60	1.80	0.91	0.06	0.01	0.97
8	0.06	0.01	0.01	0.08	0.05	<0.01	<0.01	0.07	0.02	0.00	0.00	0.02
9	0.00	0.00	0.00	0.00	<0.01	<0.01	<0.01	0.03	0.00	0.00	0.00	0.00
10	0.26	0.02	0.03	0.31	0.17	0.01	0.02	0.20	0.08	0.00	0.01	0.10
Means	0.40	0.06	0.30	0.76a	0.47	0.07	0.25	0.78a	0.39	0.06	0.10	0.54b
Desert												
1	0.27	0.02	0.07	0.36	0.24	0.06	0.09	0.39	0.10	0.03	0.03	0.16
2	0.03	0.01	0.02	0.06	0.02	0.01	0.02	0.05	0.02	0.00	0.01	0.03
3	0.02	0.01	0.03	0.06	0.02	0.01	0.02	0.05	0.02	0.00	0.02	0.05
4	0.00	0.00	0.01	0.01	0.79	0.07	0.15	1.04	0.00	0.00	0.00	0.01
Means	0.08	0.01	0.03	0.12a	0.27	0.04	0.08	0.38b	0.035	0.01	0.02	0.06a

NOTE: — = no sample analyzed.
Figures in parentheses are missing values calculated by randomized blocks missing value formula.
Means with same letter are not significantly different at the 0.05 ppm level.

FISH, WILDLIFE, AND ESTUARIES

Organochlorine Insecticide, Polychlorinated Biphenyl, and Metal Residues in Some South Dakota Birds, 1975–76 [1]

Yvonne A. Greichus, Brian D. Gueck, and Barbara D. Ammann

ABSTRACT

Chlorinated hydrocarbon insecticide, polychlorinated biphenyl (PCB), and metal residues were measured in tissues of common crows (Corvus brachyrhynchos), *American coots* (Fulica americana), *starlings* (Sturnus vulgaris), *and Franklin's gulls* (Larus pipixcan), *of South Dakota in 1975–76. Insecticides and PCBs were analyzed by column, thin-layer, and gas-liquid chromatography. Metals were analyzed by atomic absorption spectrophotometry.*

DDE was the most prevalent residue; it was detected in 93 percent of all samples and averaged 66 percent of the total residues in the carcass. Average values ranged from 0.04 ppm to 0.54 ppm. Dieldrin was detected in 61 percent of all samples and averaged < 0.01 ppm to 0.15 ppm. TDE and DDT were found in 27 percent and 15 percent, respectively, of all samples, and the averages for both ranged from < 0.01 ppm to 0.06 ppm. Heptachlor epoxide and lindane were detected in some samples. PCBs were not found above the minimum detectable level, 0.1 ppm, in any sample.

Gulls had higher insecticide and metal residues than had coots, starlings, or crows. Arsenic values averaged 1.4 ppm dry weight in carcass samples from the four species of birds. Cadmium, copper, manganese, lead, and zinc averaged 0.10, 0.94, 4.8, 1.0, and 69 ppm dry weight, respectively, and were no higher than values reported in some birds from other areas.

Introduction

Organochlorine insecticides have been used in South Dakota since 1946 for the control of noxious insects (*4*). Although many of these insecticides have been banned or limited, residues of some of the more persistent compounds such as DDT, dieldrin, and lindane are still commonly found in birds of South Dakota (*6, 7*).

Four common species of South Dakota birds with distinctly different feeding habits were analyzed in 1975–76 for eleven insecticide residues, six metals, and polychlorinated biphenyls (PCBs) to determine present levels of these chemicals so that comparisons could be made in future studies.

Methods and Materials

INSECTICIDE AND PCB ANALYSIS

Seven common crows (*Corvus brachyrhynchos*), six American coots (*Fulica americana*), six starlings (*Sturnus vulgaris*), and six Franklin's gulls (*Larus pipixcan*) were analyzed. Organochlorine insecticide and PCB residue levels were measured on a wet-weight basis in brain, liver, feather, and carcass samples from each bird. Metal levels were measured on a dry-weight basis for each bird. Samples were analyzed for lindane, heptachlor, heptachlor epoxide, dieldrin, aldrin, methoxychlor, endrin, toxaphene, DDE, TDE, DDT, zinc, cadium, lead, copper, arsenic, and manganese.

All birds were killed by shotgun. Gulls were collected September 2, 1975, approximately three miles west of Nunda, South Dakota, while feeding in a freshly plowed field. Coots were collected September 15, 1975, approximately five miles southeast of Arlington, in a marsh. Starlings were obtained February 14, 1976, near Crocker. South Dakota Game, Fish, and Parks personnel collected crows April 6, 1976, near Richmond Lake in Brown County. All specimens collected appeared to be normal and healthy.

Authors had intended to use only adults for the study but could find no literature on estimating the age of crows and starlings. They selected the seven heaviest crows for study and they analyzed all starlings collected because only six had been taken. Coots were aged by leg color (*9*) and gulls by plumage (*15*). All coots and five of six gulls analyzed were judged to be adults.

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Each specimen was necropsied to remove tissue samples and to determine sex, stomach contents, and general body condition. Technicians removed 5 g of feathers, finely cut them, and wrapped them with aluminum foil. Brains and livers were removed, weighed, and stored in glass jars. Carcass samples consisted of the entire body minus beak, legs, stomach contents, and the samples of feather, brain, and liver previously removed. After necropsy, the carcasses were wrapped and frozen in aluminum foil; several days later they were homogenized by grinding with a Toledo meat chopper, and frozen in glass jars for later analysis. All glassware used for storage and later insecticide analysis was washed in detergent, rinsed with distilled water, and baked at 425°C for at least 3 hours to remove organic contamination.

Samples were extracted and purified for chlorinated hydrocarbon residues analysis by a Florisil column method (*16*) as modified by Greichus et al. (*8*). Methods for separating PCBs and insecticides and quantitating PCBs have been described by Greichus et al. (*5*). One gram of carcass and liver and 0.5 g of brain and feathers were analyzed.

Gas chromatograph:	Varian Aerograph Model 2100
Detectors:	^{63}Ni and $Sc^{3}H$ electron-capture
Recorders:	Beckman Ten Inch, 1 mv
Columns:	6-ft × 1/16-inch borosilicate glass
Packing:	15 percent QF-1 silicone (Fluoro) or 1:1 mixture of 15 percent QF-1 and 10 percent DC-200 silicone, both on 60–100-mesh Chromosorb W (HP), acid-washed and dimethylchlorosilane-treated
Carrier gas:	Nitrogen at 40 ml/minute
Column temp.:	210° C
Injector temp.:	220° C
Detector temp.:	280° C

Identity of individual insecticides was verified by using thin-layer chromatography (*2*, *4*). Insecticides and PCBs were recovered at 89 percent and 95 percent, respectively. Minimum detection limits were set at 0.01 ppm and 0.1 ppm for insecticides and PCBs and were corrected for percent recovery but values for metals were not corrected.

METAL ANALYSIS

Zinc was determined with a Perkin-Elmer Model 303 flame atomic absorption spectrophotometer. Lead, arsenic, cadmium, copper, and manganese were determined with a Perkin-Elmer Model 503 atomic absorption spectrophotometer equipped with a heated HGA-2100 graphite furnace and a Sargent-Welch Model SRLG recorder. A Perkin-Elmer deuterium arc power supply Model 560 background corrector was used in conjunction with the spectrophotometer when necessary. Operating conditions of the instrument were essentially the same as those given by the manufacturer. Before analysis, 0.5 g dry weight of each sample was digested in 10 ml of concentrated nitric acid on a micro-Kjeldahl digestion apparatus until 2 ml of solution remained. An additional 5 ml of nitric acid was added, and the solution was boiled until 1 ml remained. Samples were reconstituted to 10 ml with distilled water and analyzed directly. Average recoveries for metals were copper 87, cadmium 91, manganese 82, arsenic 73, lead 79, and zinc, 94 percent.

Minimum detection limits used for heavy metals were 0.01 ppm for cadmium, 0.1 ppm for arsenic and lead, 0.5 ppm for copper and manganese, 1.0 ppm for zinc. In calculations of averages and totals, less than (<) values were included and given one-half the stated value; that is, a value of less than 0.1 ppm is recorded as 0.05 ppm.

Results and Discussion

INSECTICIDES AND PCBs

Average insecticide residue concentrations for common crows, starlings, American coots, and Franklin's gulls are given in Table 1. Endrin, heptachlor, methoxychlor, aldrin, and PCBs were not detected above the minimum detectable levels in any of the 100 samples analyzed. Toxaphene detected in starling feathers was judged to have been an inadvertent contaminant from a container used to carry the birds. Lindane was found in only two crows and was not used in the calculation of average total insecticides. One crow had carcass and liver residues of 0.01 ppm and 0.11 ppm lindane, respectively; another crow had a carcass residue of 0.01 ppm. Heptachlor epoxide was detected in crow carcass and liver samples and in one crow brain. Dieldrin residues were found in all species, all four tissue types, and in 61 percent of all samples, except the coot. Dieldrin was either absent from the tissues of the coot, or present in the liver at the limit of detection, 0.01 ppm. Dieldrin concentrations in the brain and feathers of the four species were usually below or slightly above the 0.01 ppm lower analytical limit.

DDT and its metabolites were the residues found most consistently. DDE was the most prevalent of the DDT complex and was found in 93 percent of all samples. TDE and DDT were detected in 27 percent and 15 percent, respectively, of all samples.

Starlings reflect the general environmental levels of organochlorine insecticides and metals available to them in South Dakota because they are often year-around terrestrial residents. Coots and Franklin's gulls do not reflect true South Dakota contamination levels because they are summer resident only and are subject to migratory contamination in other areas. The low levels of TDE and DDT may reflect the decreased use and eventual banning of DDT in

TABLE 1. *Organochlorine insecticide residues in South Dakota birds, 1975–76*

Bird	Average Residues, ppm (μg/g) Wet Weight					
	Heptachlor Epoxide	Dieldrin	DDE	TDE	DDT	Total Insecticides
CARCASS						
Crow	0.06	0.13	0.54	0.04	0.06	0.84
Coot	<0.01	<0.01	0.04	<0.01	<0.01	0.06
Starling	<0.01	0.02	0.06	<0.01	<0.01	0.10
Gull	<0.01	0.15	0.44	0.06	<0.01	0.66
LIVER						
Crow	0.10	0.05	0.41	0.04	0.02	0.61
Coot	<0.01	0.01	0.02	<0.01	<0.01	0.05
Starling	<0.01	0.04	0.06	<0.01	<0.01	0.12
Gull	<0.01	0.04	0.10	0.04	<0.01	0.20
BRAIN						
Crow	<0.01	0.07	0.05	<0.01	<0.01	0.13
Coot	<0.01	<0.01	0.01	<0.01	<0.01	0.04
Starling	<0.01	<0.01	0.02	<0.01	<0.01	0.04
Gull	<0.01	<0.01	0.02	<0.01	<0.01	0.04
FEATHERS						
Crow	<0.01	0.06	0.04	<0.01	<0.01	0.11
Coot	<0.01	<0.01	0.02	<0.01	<0.01	0.04
Starling[1]	—	—	—	—	—	—
Gull	<0.01	0.02	0.05	<0.01	<0.01	0.08

Note: Seven crows and six each of coots, starlings, and gulls were analyzed.
[1] Starling feathers were contaminated with toxaphene at bird-collection site; no residues are reported here.

the United States in 1973, although DDE is still common in the environment.

Nationwide monitoring of mallard and black duck wings by the Fish and Wildlife Service, U.S. Department of the Interior, since 1965 has shown DDE to the the predominant residue (*10, 11*). Results of the monitoring in 1965–66 showed DDE to be the predominant residue, followed by DDT, TDE, dieldrin, and heptachlor expoxide; in 1969, DDE was followed by PCBs, DDT, dieldrin, TDE, and heptachlor epoxide. In both studies, organochlorine residues were generally highest in the Atlantic and Pacific flyways and lowest in the Central flyway of which South Dakota is a part, and in the Mississippi flyway.

Total insecticide residues were consistently higher in crows than in other species. Franklin's gulls had the second highest total residue level, followed by starlings and American coots. In brain samples, however, all three species had approximately equal concentrations. Carcass samples usually had the highest insecticide levels, followed by livers; brains and feathers were about equal.

Martin (*13*) analyzed carcasses of starlings from 128 areas of the United States in 1967–68 and found DDT, its metabolites, and dieldrin in all sites. At four South Dakota sites, the average residues for 1967–68 ranged from 0.103 ppm to 1.925 ppm DDE, 0.013 ppm to 0.018 ppm TDE, 0.018 ppm to 0.030 ppm DDT, and 0.012 ppm to 0.080 ppm dieldrin. Heptachlor epoxide and lindane were occasionally found at all South Dakota sites. Average total insecticide residues were 0.234, 0.201, 2.054, and 0.334 ppm at the four sites (*13*). Starlings monitored for the present study in 1976 had lower average total insecticides, 0.10 ppm, than had birds in any of the four South Dakota sites studied by Martin (*13*).

METALS

Average concentrations of metals in carcasses of crows, coots, gulls, and starlings are reported in Table 2. Values are given on a dry-weight basis but can be converted to the approximate wet weight by multiplying the value by 0.43, which was the average dry weight of 1 g of bird carcass. Arsenic levels were similar in all four types of birds. Converted to wet weight, arsenic residues were greater than those reported by Martin and Nickerson (*14*). Starlings collected from 50 sites in the United States contained < 0.05 ppm wet weight arsenic except for one sample with 0.21 ppm arsenic (*14*). Gulls averaged 0.21 ppm cadmium, higher than residues in other birds of this study but lower than some values reported for starlings by Martin and Nickerson (*14*).

TABLE 2. *Metal residues in South Dakota bird carcasses, 1975–76*

Metal	Average Residues, ppm (μg/g) Dry Weight[1]			
	Gulls	Coots	Starlings	Crows
Arsenic	1.6	1.5	1.6	1.0
Cadmium	0.31	0.08	0.10	0.03
Copper	1.8	0.75	0.51	0.75
Manganese	4.5	9.8[2]	4.0	4.2
Lead	3.2[2]	0.86	0.77	0.72
Zinc	82.0	71.0	75.0	52.0

Note: Seven crows and six each of coots, starlings, and gulls were analyzed.
[1] Residues can be converted to wet weight by multiplying each value by 0.43, the average dry weight of 1 g of bird carcass.
[2] Two birds were analyzed.

Gulls also had higher concentrations of lead than had coots, starlings, or crows. The gulls could have been contaminated in areas other than South Dakota because they are migratory. A possible cause could be the ingestion of shot. Waterfowl are susceptible to shot ingestion in wetland areas; upland birds are susceptible to a lesser extent in terrestrial areas (*1*). Lead residues in South Dakota starlings averaged 0.36 ppm in 1971 (*14*), which is close to 0.33 ppm wet weight found among starlings in the present study.

Manganese, copper, and zinc are essential dietary elements and are not usually considered contaminants. Levels of copper and zinc (Table 2) reported for the four types of birds were not unusual. Considerably higher levels of copper (21 ppm wet weight) and zinc (76 ppm wet weight) have been found in livers of white pelicans (*12*). Manganese concentrations of 9.8 ppm were more than twice as great in coots than in other birds, possibly because their diet contains aquatic plants rich in this element. Some aquatic plants have comparatively high levels of manganese (660 ppm dry weight) (*3*).

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Organochlorine Pesticide Residues in Florida Birds of Prey, 1969–76

David. W. Johnston [1]

ABSTRACT

Chlorinated hydrocarbon pesticide residues, especially DDT and its metabolites, were determined in 71 individuals of 14 species of predatory birds obtained in Florida between 1969 and 1976. Of the 71 birds, 68 contained p,p'*-DDE or another DDT metabolite; 34 contained dieldrin. DDE was found in 93 percent of the 57 adipose tissue samples, all the 9 brain samples, and 89 percent of the 62 uropygial gland samples. Of the 65 birds taken since 1972, 61 contained DDE in at least one of these three tissues. The annual average of* Σ*DDT in adipose tissue and uropygial gland over the 6-year span was approximately 5 ppm wet weight. From 1973 to 1976, no significant increase or decrease in pesticide burdens was detected. Some birds had no DDE whereas others contained up to 76 ppm* Σ*DDT. None of the data suggest that any of the birds of prey had died of DDT or DDT metabolite poisoning.*

Introduction

For approximately two decades in North America, much public and scientific interest has been focused on population declines of various birds of prey including eagles, osprey (*Pandion haliaetus*), and peregrine falcon (*Falco peregrinus*). In some species, correlations have been made or suspected between pesticide burdens, especially DDE, and mortality, population declines, or altered physiological processes resulting in impaired reproductive performances (*8, 18*). Eggshell thinning is now believed to be a result of high DDE burdens, both in captive and feral birds of prey (*13, 14, 17*). One might anticipate high pesticide burdens in birds of prey because they are usually terminal members of food chains, and thus can concentrate the fat-soluble chlorinated hydrocarbon pesticides. In most published accounts dealing with these birds, pesticide residues were extracted from eggs or nestling birds or from birds experimentally fed DDT (*4, 13, 14*); there are few published accounts of body burdens in adults except for a limited number of autopsied birds found dead and suspected of pesticide poisoning. In fact, virtually nothing has been published on body burdens in feral adult birds of prey which reportedly produced thin eggshells. Thus, to date, pesticide burdens at levels presumably not impairing reproduction are poorly documented (*2*). In the present report, some organochlorine pesticide residues extracted from birds of prey obtained recently in Florida are quantitated.

Sampling Methods

The birds analyzed were obtained between 1969 and 1976, chiefly in northcentral Florida near Gainesville. Most birds were fresh roadkills or were illegally shot by hunters. A few were picked up alive in a weakened condition or were having convulsions; they were kept in an aviary, and died within 24 hours. With the possible exception of the latter birds, the present report includes birds dying accidentally, that is, there was no a priori suggestion that any pesticide burden contributed to death.

The sample includes two orders (Falconiformes: vultures, kites, hawks, falcons, osprey, caracara; Strigiformes: owls). In all, 6 families, 12 genera, 14 species, and 71 individuals were analyzed.

Analytical Procedures

From each specimen, whether fresh or previously frozen in individual plastic bags, samples of subcutaneous adipose tissue (fat) and/or the entire uropygial gland and/or the cerebrum were removed for analysis. Recently, a number of investigators have indicated the possibility of using the unique avian uropygial, or preen, gland as an indicator of pesticide burdens in birds (*3, 4, 11*). In feral, migratory songbirds, Johnston (*11*) reported a high correlation,

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$r = 0.7568$, of ΣDDT between adipose tissue and uropygial gland. In the present study, birds varied in the degree of obesity; in some, essentially no fat could be located, so only the gland or brain was used for analysis. For 59 samples of fat, the mean sample weight was 1.1224 g; for 62 samples of uropygial glands, the mean weight was 0.5590 g; and the mean brain weight taken from 9 birds was 4.1375 g. Each sample was individually thoroughly homogenized in sodium sulfate, and extracted for at least 12 hours with petroleum ether in a Soxhlet apparatus. The lipid extract was evaporated to dryness, weighed, and partitioned with acetonitrile and hexane.

All samples were analyzed on a Model 600-D Varian gas chromatograph with the following instrument parameters and operating conditions:

Detector:	electron-capture
Column:	6-ft × ¼-inch glass, packed with a mixture of 6.4 percent OV-210 and 1.6 percent OV-17 (1+1) on Chromosorb W
Temperatures:	injection port 210° C column 212° C detector 215° C
Carrier gas:	nitrogen flowing at 45 ml/minute

Recoveries for the organochlorine compounds ranged from 75 percent to 95 percent. Sensitivity was approximately 0.01 ppm.

Results

Table 1 contains the results of analyses for the 71 birds of prey. Tissues analyzed for the individual birds were not

TABLE 1. *Chlorinated pesticide burdens in Florida birds of prey, 1969–76*

County	Date	Sex Age [1]	Tissue [2] and Sample Weight, g	Residues, ppm Wet Weight		
				p,p'-DDE	ΣDDT	Dieldrin
CATHARTES AURA (TURKEY VULTURE)						
Alachua	Nov. 71	UNK	A (2.0751)	3.37	3.55	0
Levy	Apr. 73	M	A (3.0238)	1.32	3.00	0.07
			B (6.0752)	0.17	0.17	0
Alachua	Apr. 73	F	A (4.0514)	0.78	1.32	0.46
			B (5.5159)	0.11	0.11	0
Levy	May 73	M	A (2.5833)	1.59	2.28	0.07
			B (6.1278)	0.01	0.01	0
Levy	May 73	M	A (2.6192)	2.86	4.21	0.19
			B (4.7010)	0.04	0.04	0
Gilchrist	May 73	M	A (1.9826)	4.04	5.05	0.16
			B (5.7562)	0.69	0.87	0.02
Leon	July 74	F	A (1.8175)	3.03	3.74	0.74
			U (0.3899)	0.64	1.03	0
Leon	Sept. 74	M	A (1.9901)	1.18	1.41	0.05
			U (0.3023)	0	0	0
Leon	Oct. 74	F	A (1.4910)	9.05	13.35	0.30
			U (0.3417)	6.44	6.44	0
Leon	Oct. 74	F	A (1.5841)	7.89	9.72	1.11
			U (0.4800)	5.00	5.00	0.16
CORAGYPS ATRATUS (BLACK VULTURE)						
Alachua	Jan. 72	M	F (2.0323)	6.39	11.75	1.18
Marion	May 73	F	A (2.8724)	3.83	6.87	0.22
			U (1.0003)	10.50	15.25	0.50
Alachua	May 73	F	A (2.2551)	1.26	1.49	0.04
			B (5.6169)	0.10	0.11	0

(Continued next page)

TABLE 1 (continued). *Chlorinated pesticide burdens in Florida birds of prey, 1969–76*

County	Date	Sex/ Age [1]	Tissue [2] and Sample Weight, g	Residues, ppm Wet Weight		
				p,p'-DDE	ΣDDT	Dieldrin
			***CORAGYPS ATRATUS* (BLACK VULTURE)—Continued**			
Alachua	May 73	M	A (3.0264)	3.06	7.68	0.66
			U (0.8266)	5.44	6.84	0.24
Alachua	June 74	M	A (0.9518)	11.56	25.43	2.02
			U (0.8682)	9.33	14.74	0.69
			***ELANOIDES FORFICATUS* (SWALLOW-TAILED KITE)**			
Marion	May 75	M	U (0.6269)	0.48	0.48	0
			***ACCIPITER STRIATUS* (SHARP-SHINNED HAWK)**			
Alachua	Nov. 75	F	A (0.3009)	16.62	17.12	0
			U (0.0637)	19.62	19.62	0
			***ACCIPITER COOPERII* (COOPER'S HAWK)**			
Alachua	Sept. 73	F	U (0.2763)	12.12	13.02	0
			***BUTEO JAMAICENSIS* (RED-TAILED HAWK)**			
Alachua	Jan. 73	IM	A (1.8650)	6.25	6.25	0
			U (0.4830)	0.10	0.10	0
Alachua	Jan. 73	IMF	A (1.0422)	3.49	3.73	0.89
			U (0.4788)	0.11	0.11	0
Alachua	July 73	IM	A (0.7617)	0.37	0.48	0.04
			U (0.7570)	0.39	0.50	0
Alachua	July 74	IM	A (1.6963)	0.59	1.32	0
			U (0.4846)	0.21	0.21	0
Alachua	Dec. 74	F	A (0.5357)	0.37	0.37	0
			U (0.4527)	0	0	0
Alachua	Jan. 76	M	A (0.4694)	4.26	5.86	0.85
			U (0.3936)	0.38	0.38	0
Madison	Jan. 76	IM	A (0.5699)	6.14	7.19	2.37
			U (0.2852)	1.05	1.05	0.18
			***BUTEO LINEATUS* (RED-SHOULDERED HAWK)**			
Alachua	Jan. 72	F	A (1.3596)	0.64	1.04	0.06
			U (0.2853)	0.18	0.18	0
Alachua	Sept. 73	IM	A (0.3310)	0.45	1.21	0
			U (0.3104)	0.24	0.60	0
Alachua	Jan. 76	AD	A (0.1888)	7.15	7.15	0
			U (0.2552)	0.39	0.39	0
Pinellas	Jan. 76	F	A (0.2615)	34.42	61.76	38.24
			U (0.2514)	0.80	1.80	0.80
Baker	May 76	F	U (0.6186)	1.21	1.21	0

(Continued next page)

TABLE 1 (continued). *Chlorinated pesticide burdens in Florida birds of prey, 1969–76*

County	Date	Sex/ Age [1]	Tissue [2] and Sample Weight, g	Residues, ppm Wet Weight		
				p,p'-DDE	ΣDDT	Dieldrin
			PANDION HALIAETUS (OSPREY)			
Marion	—	JUV	A (0.3864)	0.13	0.26	0
			U (2.4581)	1.55	1.79	0
Monroe	Apr. 73	JUV M	A (2.8327)	0.55	1.29	0
			U (4.1778)	0.41	0.62	0
Pinellas	Sept. 74	UNK	A (0.3840)	0	0	0
			U (1.1643)	0.09	0.09	0
Pinellas	Oct. 74	M	A (0.7767)	0.33	0.33	0
			U (0.6309)	0.32	0.32	0
Alachua	Apr. 75	F	U (0.4922)	13.21	15.85	0.91
Pinellas	May 76	F	A (0.4861)	1.65	1.87	0
			U (3.7545)	0.53	0.71	0
Pinellas	May 76	M	A (0.4264)	1.52	2.46	0
			U	1.39	1.39	0
			CARACARA CHERIWAY (CARACARA)			
Glades	July 75	AD F	A (0.1416)	2.47	2.47	0
			U (0.5854)	1.25	1.25	0
Highlands	July 75	IM F	A (0.0805)	1.24	1.24	0
			U (0.6235)	0.48	0.48	0
Highlands	Apr. 76	IM F	A (0.1229)	3.25	3.25	0
			U (0.5734)	2.44	2.44	0
			FALCO SPARVERIUS (AMERICAN KESTREL)			
Indian Riv.	Mar. 73	M	A (0.2810)	14.59	16.37	0.36
			U (0.0954)	3.15	3.15	0
Indian Riv.	Mar. 73	F	A (1.1978)	1.77	2.09	0
Indian Riv.	Mar. 73	F	A	20.57	22.71	0
			U	4.63	4.63	0
Broward	Mar. 73	F	A (0.1588)	0.79	0.79	0
			U (0.0788)	0.63	0.63	0
Indian Riv.	Mar. 73	F	A (0.1399)	2.14	2.14	0
			U (0.0602)	1.66	1.66	0
Indian Riv.	Mar. 73	F	A (0.0551)	9.07	9.07	0
			U (0.0833)	1.80	1.80	0
Indian Riv.	Mar. 73	M	U (0.0653)	0	0	0
Indian Riv.	Jan. 74	M	A (0.0980)	0	0	0
			U (0.0258)	1.94	1.94	0
Indian Riv.	Oct. 74	M	A (0.0262)	0	0	0
			U (0.0302)	0	0	0
Leon	Jan. 75	M	A	7.61	8.03	0
			U	1.44	2.20	0
Pinellas [3]	Nov. 75	M	U (0.0486)	4.12	4.12	3.09
			B (1.0756)	0.42	0.42	0.70
Pinellas [3]	Nov. 75	M	U (0.0459)	7.63	18.53	4.36

(Continued next page)

TABLE 1 (continued). *Chlorinated pesticide burdens in Florida birds of prey, 1969–76*

County	Date	Sex/ Age [1]	Tissue [2] and Sample Weight, g	Residues, ppm Wet Weight		
				p,p'-DDE	ΣDDT	Dieldrin
FALCO SPARVERIUS (AMERICAN KESTREL)						
			B (1.1574)	0.30	0.74	0.82
Pinellas [3]	Nov. 75	F	U (0.0486)	2.06	2.06	1.03
			B (1.2114)	1.03	1.03	0.37
Pinellas [3]	Feb. 76	M	A (0.0790)	0	0	0
			U (0.0505)	0	0	0
TYTO ALBA (BARN OWL)						
Indian Riv.	Mar. 69	M	A (0.9538)	8.28	9.27	1.68
			U (2.1913)	1.31	1.31	0
Indian Riv.	May 76	M	U (0.3550)	0	0	0
OTUS ASIO (SCREECH OWL)						
Alachua	Oct. 71	F	A (1.1317)	6.19	6.19	0
			U (0.1201)	1.17	1.17	0
Levy	Sept. 73	UNK	A (0.3585)	0.26	0.26	0
			U (0.1454)	0	0	0
Alachua	Dec. 75	UNK	A (0.6573)	0.30	0.30	0
			U (0.1435)	3.48	3.48	0
Indian Riv.	May 75	UNK	U (0.0378)	10.58	10.58	0
Pinellas [3]	Jan. 76	M	U (0.0502)	49.80	49.80	0
Pinellas [3]	May 76	M	U (0.0748)	1.34	1.34	0
BUBO VIRGINIANUS (GREAT HORNED OWL)						
Leon	Mar. 73	UNK	A (3.0278)	2.06	3.59	0.08
			U (0.6823)	8.24	8.68	0
Alachua	Nov. 75	F	A (0.6460)	5.42	9.21	3.02
			U (0.7466)	3.28	4.28	0.47
Dixie	Dec. 75	UNK	U (0.4400)	17.05	17.05	0
Marion	May 76	MF	A (0.2821)	9.75	12.05	6.20
			U (0.5537)	0.81	0.81	0
STRIX VARIA (BARRED OWL)						
Dixie	Apr. 73	UNK	A (1.5201)	5.76	6.90	0.21
			U (0.7385)	5.08	5.69	0.14
Alachua	May 74	F	U (0.5460)	74.18	76.93	0
Alachua	Dec. 75	F	A (0.2286)	1.09	1.09	0
			U (0.5321)	0.37	0.37	0.40
Leon	Jan. 75	M	A (0.1183)	7.61	8.03	0
			U (0.6588)	1.44	2.20	0
Pasco	Mar. 76	AD	A (0.1830)	1.09	1.09	0
			U (0.5843)	0.73	0.73	0

[1] M=adult male, F=adult female, AD=adult of undetermined sex, UNK=bird of unknown sex or age, JUV=juvenile, IM=immature. Sex of juvenile and immature birds was not always recorded.
[2] Tissue abbreviations: A=adipose tissue, U=uropygial gland, B=brain.
[3] Birds that reportedly died in captivity and exhibited convulsions.

always perfectly uniform because birds were obtained in different ways by different persons, and it was frequently inconvenient or impossible to take samples of brain, fat, and the uropygial gland from every bird. Furthermore, due to its relatively superficial position, the uropygial gland was sometimes damaged, and quite often a specimen was so lean that no fat could be found for pesticide analysis.

Even so, a number of important features emerge from the data in Table 1. All taxa (family, genus, and species) had some birds containing *p,p'*-DDE or other DDT metabolite. Dieldrin, on the other hand, was not present in all taxa. Of the 71 birds, 68 (96 percent) contained DDE but only 34 (48 percent) contained dieldrin in at least one of the three tissues. In the three tissues analyzed 93 percent of the fat samples contained DDE, 100 percent of the brains contained DDE, and 89 percent of the uropygial glands contained DDE. These values indicate a nearly universal occurrence of DDE in the birds studied and in the three tissues sampled.

In the 45 birds of prey in which both adipose tissue and uropygial gland were analyzed and in which one or both samples contained DDE, 40 (89 percent) had DDE in both tissues, 3 (7 percent) had DDE in adipose tissue only, and only 2 (4 percent) had DDE in the uropygial gland alone. However, Figure 1 shows a poor correlation ($r = 0.3398$) of ΣDDT, in ppm wet weight, between these two tissue types. Of 46 birds, 40 had higher concentrations of ΣDDT in the adipose tissue than in the uropygial gland. For the species analyzed, the mean ratio of ΣDDT in adipose tissue to uropygial gland was 2.6:1, a higher ratio than the 2.2:1 reported by Johnston (*11*) for a sample of other feral species such as loons, cormorants, herons, and gulls.

Figures 2 and 3 show ΣDDT found, respectively, in the adipose tissue and uropygial gland through the sampling period. In both samples, median values were calculated for all the species in a given year; these values are indicated by a solid line. The lines might not indicate realistic trends because the numbers of a given species available for analysis varied from year to year, as indicated by raw data in Table 1.

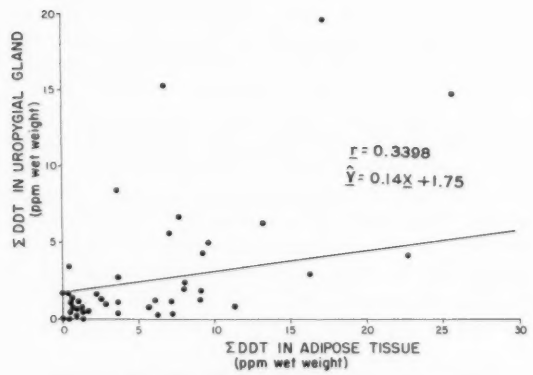

FIGURE 1. *Relationship of ΣDDT in adipose tissue and uropygial gland in Florida birds of prey, 1969–76*

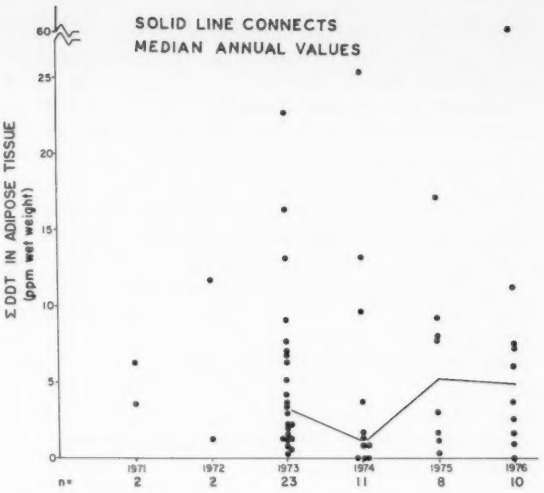
SOLID LINE CONNECTS MEDIAN ANNUAL VALUES
Σ DDT IN ADIPOSE TISSUE (ppm wet weight)
1971 1972 1973 1974 1975 1976
n= 2 2 23 11 8 10

FIGURE 2. *ΣDDT in adipose tissue of Florida birds of prey, 1971–76*

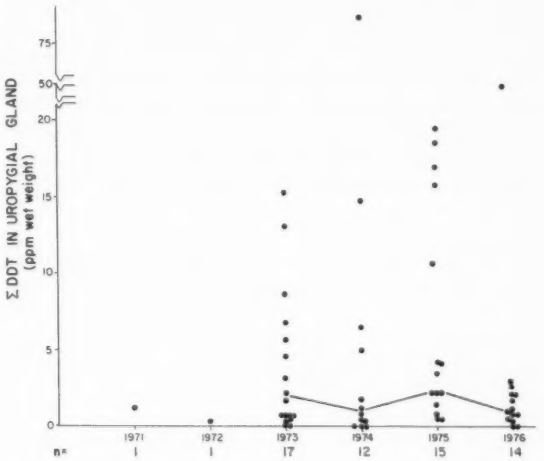

FIGURE 3. *ΣDDT in uropygial glands of Florida birds of prey, 1971–76*

Discussion

Of the 14 species examined here, there is no assurance that any bird was a permanent resident where collected except for the caracaras and juvenile osprey. Any or all the birds might have been transient or migratory at some time during their lives, so pesticide burdens determined for these birds

were not necessarily accumulated in Florida, but could have come from prey consumed on a wintering area south of the state, from a breeding area in the north, or in intervening areas during migratory flights. It is likely, however, that most of the four owl species were Florida residents because they tend to migrate less than the other birds of prey studied here.

Subspecific information was determined only for the American kestrel. These small falcons were all representative of the northern subspecies (*Falco s. sparverius*) which migrates into Florida from the northern United States and winters in large numbers in the state. The author was unable to obtain samples of the local resident Florida subspecies, *F. s. paulus*, for analysis. Data on the vultures, *Cathartes* and *Coragyps*, are presented in Table 1 and appear to be the first residue findings published on these species.

Because vultures are terminal members of food chains, they might be expected to have exceptionally high DDT levels, but this does not appear to be true of *Cathartes*. Only 1 of 10 turkey vulture fat samples exceeded 10 ppm ΣDDT; the mean was 4.76 ppm. However, the mean for the fat of 5 *Coragyps* was 10.64 ppm ΣDDT; one bird had 25.43 ppm. Both vultures scavenge road-killed animals such as the nine-banded armadillo, Virginia opossum, dogs, and various smaller mammals, birds, and reptiles in Florida, most of which are nonmigratory. Why *Coragyps* should have a higher mean burden of ΣDDT than do *Cathartes* is unclear.

The *Accipiter* hawks, also called bird hawks, have pesticide burdens as high as or higher than most other species studied (Table 1). The small sample size precludes generalizations, but it is noteworthy that in 1972 Henny reported that the Cooper's hawk "is in serious jeopardy in the northeastern U.S." (*6*).

Some species listed in Table 1 are largely insectivorous (*5*), namely, *Elanoides forficatus*, *Falco sparverius*, and *Otus asio*. At least three of the 14 specimens of *Falco* had ΣDDT burdens exceeding 10 ppm; one contained 18.53 ppm in the uropygial gland, and the ΣDDT burden in adipose tissue probably exceeded 50 ppm. Two of six *Otus* specimens had exceptionally high levels in their uropygial glands: 10.58 ppm and 49.80 ppm. For this species, 10 ppm DDE dry weight in the diet produced thin eggshells (*13*). Although dietary levels of DDT may not be directly related to levels in the adipose tissue or uropygial gland, it is significant that 5 ppm DDT wet weight in the diet of *Falco sparverius* resulted in the classical eggshell-thinning syndrome (*17*). However, carcass (*17*) and breast muscle (*8*) analyses of dead or dying American kestrels in the northern United States had generally higher DDT burdens than those found in the present study (*1*). For this species, it is significant that three individuals contained no DDT or metabolite (Table 1).

Henny et al. presented data on eggshell thicknesses and populations of red-shouldered hawks (*Buteo lineatus*) from a refuge in Maryland (*7*). The authors thought it "doubtful that the relatively low pesticide levels in the eggs had a detrimental effect on the reproductive performance of the population." Except for a single bird containing 61.76 ppm ΣDDT and 38.24 ppm dieldrin, organochlorine residues in this species were generally low (Table 1).

The osprey (*Pandion haliaetus*) was studied intensively in the 1960s because its population had declined precipitously in some areas (*6*). As with other species discussed in this paper, presticide levels in eggs and nestlings have been published but data for adults are scarce. Wiemeyer et al. reported brain and carcass analyses of dead birds in Connecticut and Virginia (*18*). DDE residues in carcasses averaged 23 ppm wet weight, generally, exceeding the levels in adipose tissue and uropygial gland in Florida birds reported here. Because different tissues were analyzed, it is difficult to compare previously published data on red-tailed hawks and great horned owls with those reported here. For three nestling red-tailed hawks, Seidensticker found an average of 21.50 ppm ΣDDT wet weight, in breast muscle (*15*). Seidensticker and Reynolds reported 1.40 ppm wet weight ΣDDT in nestling red-tail hawk muscle and 9.29 ppm ΣDDT in the muscle of a great horned owl (*16*).

Two generalities emerge from the data in Table 1 and Figures 2 and 3. There is no firm evidence for this sample of birds of prey from Florida that DDE and dieldrin burdens diminished in 1971–76. In both the adipose tissue and uropygial glands, the data indicate an approximate average of 5 ppm over the 4–6 year span. Small migratory songbirds, on the other hand, showed a dramatic decrease of DDE in adipose tissue from 1964 to 1973 (*9*, *10*). That decrease was correlated with the decreased use of DDT in the United States during the same time. Presumably, the ban on DDT use in the United States imposed by the U.S. Environmental Protection Agency (EPA) December 31, 1972, should have reduced the amount of DDT in natural ecosystems. The birds of prey studied here are significant, especially those analyzed after 1972, because a large proportion did contain DDT or a metabolite. How would a hawk, owl, or vulture obtain significant quantities of DDT in 1976? It is plausible that a long-lived bird could have accumulated small pesticide quantities for years and simply stored them in adipose tissue. Unless these deposits were totally depleted for energy resources, the pesticides might not have been mobilized into the bird's bloodstream or eliminated except in very small quantities. The data on uropygial glands presented in Table 1 indicate that birds of prey eliminate smaller quantities of pesticides through this gland than do other types of birds (*3*, *4*).

A second possible explanation for the DDT burden in birds of prey after the EPA ban in 1972 is that at least eight species analyzed here might have migrated to Florida from

the West Indies or Central America where they could have obtained DDT-contaminated foods. This is probably similar to the situation of the migratory American kestrels discussed by Lincer and Sherburne *(12)*. They suggested that this species obtained pesticide-laden foods chiefly from the wintering grounds rather than from nesting sites in the northern United States. They state: "The disastrous role played by the far-removed, but inordinately contaminated, winter prey once again dramatically points out the global nature of the biocide problem." Still, the presence of DDT in tissues of the caracara, which is a resident of southcentral Florida, is an enigma.

Since 1973, the ΣDDT burdens in adipose tissue of two species examined here, osprey and American kestrel, were very low (0–2 ppm).

Acknowledgments

O. L. Austin, Jr., P. Brodkorb, R. L. Crawford, D. J. Forrester, T. Gilyard, R. Heath, Jr., H. W. Kale, II, S. A. Nesbitt, and J. M. Whittier assisted in collecting birds. Birds from Leon County, Florida, were provided by personnel at the Tall Timbers Research Station. I thank the following for help in laboratory analyses: R. Bull, G. Gause, A. Meylan, and M. Raum. An earlier draft of the manuscript was critically examined by W. H. Stickel. Illustrations were prepared by E. Belcher.

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Shell Thinning and Pesticide Residues in Texas Aquatic Bird Eggs, 1970

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ABSTRACT

Significant decreases in eggshell thickness were found in 15 of 22 species of aquatic birds in Texas in 1970. Shell thickness reductions of 9 to 15 percent were found in white pelicans (Pelecanus erythrorhynchos), *brown pelicans* (P. occidentalis), *and great blue herons* (Ardea herodias). *DDT family compounds were found in all eggs, and mean residues ranged from 0.4 ppm in white ibis* (Eudocimus albus) *to 23.2 ppm in great egrets* (Casmerodius albus). *ΣDDT residues were negatively correlated with shell thickness in five species; PCBs were negatively correlated in two. Residues in marine birds were generally lower and more uniform than levels in birds feeding in fresh and brackish water. DDT and dieldrin residues were higher in eggs from colonies near agricultural areas where these insecticides were heavily used; higher PCB residues were consistently associated with urban and industrial areas. Populations of five species have declined and deserve continued study: brown pelican, reddish egret* (Dichromanassa rufescens), *white-faced ibis* (Plegadis chihi), *laughing gull* (Larus atricilla), *and Forster's tern* (Sterna forsteri). *Population trends of four other species were undetermined and should be followed closely in future years.*

Introduction

Eggshell thinning has been noted in a number of declining populations of fish-eating birds in the United States (*2, 6, 19, 20*). Laboratory investigations show that the DDT family compounds, ΣDDT, primarily DDE, induce shell thinning in some wild birds and their eggs (*15, 16, 28*). The recent decline in brown pelicans, reddish egrets, and an apparent decline in white-faced ibis on the Texas Gulf Coast prompted the present study to determine the extent of eggshell thinning and the impact of pesticide contamination on these and other fish-eating birds breeding in Texas. The authors present information on shell thickness changes and chemical residues in eggs of 22 species of aquatic birds. Sources of contamination and species threatened by exposure to pesticides are identified.

Study Area and Methods

From March through July 1970, 1,043 eggs were collected in 30 locations on the Texas Coast. One egg was taken randomly from each nest sampled in a pattern distributed as evenly as possible throughout each colony. Whole eggs were weighed and measured, wrapped in aluminum foil, and frozen. Contents were later removed, stored in jars prerinsed with acetone, and immediately refrozen until analysis. Five to 20 eggs of each species were analyzed at the Denver Wildlife Research Center Laboratory, Denver, Colorado. Chemical analyses were completed in 1970 and 1971. Except for brown pelican eggs which were addled, only fresh eggs were analyzed for pesticide residues.

The authors biased selection of eggs for chemical analysis by singling out thin-shelled eggs from each species. Random samples of white-faced ibis, black-crowned night heron, and Forster's tern eggs were also analyzed. Mercury levels were determined in 10 white-faced ibis and 10 great blue heron eggs.

Organochlorine residues and polychlorinated biphenyls (PCBs) were determined by using methods described by Peterson et al. (*25*). The methods measure ΣDDT, aldrin, dieldrin, endrin, heptachlor epoxide, and lindane at 0.1 ppm wet weight, and chlordane and toxaphene at 0.5 ppm wet weight. PCBs were not separated from pesticides before measurement. When found, PCBs were identified on two separate columns and by visual comparison of chromatograms with standard Aroclors. The PCB residues were quantitated by averaging peak responses and comparing them with Aroclor 1254 standards. Detection limit of the procedure for PCBs was 0.5 ppm. Mercury residues

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were determined by using methods described by Okuno et al. (*24*). No corrections were made for possible moisture loss.

The authors compared shell thicknesses of eggs collected in 1970 with those of museum eggs collected before widespread use of DDT. Data on white and brown pelican eggshells collected before 1947 are from Anderson and Hickey (*2*); data on white-faced ibis eggshells were supplied by A. J. Smith and J. O. Keith (personal communication, 1971). All other measurements of eggshells collected before 1943 were obtained from the Western Foundation of Vertebrate Zoology, Los Angeles, California, and Welder Wildlife Foundation, Sinton, Texas. Anderson and Hickey (*1*), showed that eggshell thickness for a particular species varies significantly over broad geographic areas particularly with latitude. Whenever possible, museum eggs from the Texas Coast and other southern latitudes were selected for shell thickness measurement.

Results and Discussion

EGGSHELL CHANGES

Fifteen of 22 species sampled showed a signficant negative change in eggshell thicknesses from their museum mean (Table 1). The species with the greatest average thinning were the white pelican (15 percent), great blue heron (13 percent), and brown pelican (11 percent). No collapsed eggs were found in the nests of these species. Although the average thinning of white-faced ibis eggshells was only 4 percent, numerous collapsed, dented, and cracked eggs were found in and around ibis nests. In 1971, continued sampling showed that about 3.5 percent of the white-faced ibis eggs in marked nests had dented or cracked shells; the incidence of cracked eggs of other species was less than 1 percent. Numerous field studies have shown that eggshell thinning of less than 10 percent seldom incurs egg breakage (*3, 6, 10*). Egg loss becomes evident with thinning of 10–15 percent (*19*), and serious breakage, usually accompanied by population decline, occurs when eggshell thinning exceeds 15 percent (*2, 20*). The degree of shell thinning among the white and brown pelicans and great blue heron approaches that found in other populations in which shell thinning adversely affected reproduction.

Average shell thinning was greatest in the Lower Laguna Madre–Green Island region (Figure 1). Shell thickness did not vary significantly among heronries sampled elsewhere on the Texas Coast.

ORGANOCHLORINE RESIDUES

Residues of ΣDDT, primarily DDE, were found in all samples. The highest averages were in eggs of the great egret, 23.2 ppm; Caspian tern, 15.1 ppm; and laughing gull, 10.4 ppm (Table 2). ΣDDT in the eggs of the remaining species ranged from 0.4 ppm in white ibis to 9.7 ppm in black skimmer. Consistently higher levels of ΣDDT and the greatest amount of shell thinning were found in eggs from the lower coast near the intensively cultivated Rio Grande Valley. ΣDDT compounds were found in eggs of species that feed in all habitats: freshwater, brackish, and marine.

Dieldrin residues, found in 14 species, were highest in the snowy egret, white-faced ibis, and great egret (Table 2), species that feed primarily in freshwater and brackish marshes. Little dieldrin was found in eggs of ocean-feeding birds such as brown pelican, royal tern, and Sandwich tern. Greatest dieldrin residues were in eggs from colonies adjacent to the Texas rice belt where aldrin had often been used to treat rice seed.

TABLE 1. *Eggshell changes of several Texas fish-eating birds, pre-1943 and in 1970*

Species	Period[1,2]	No.	Shell Thickness, mm Mean±SE	Change, %
White Pelican	pre-1947	102	0.676±0.005	
Pelecanus erythrorhynchos	1970	28	0.577±0.008[3]	−15
Great Blue Heron	pre-1943	32	0.413±0.005	
Ardea herodias	1970	74	0.359±0.003[3]	−13
Brown Pelican	pre-1947	43	0.557±0.006	
P. occidentalis	1970	14	0.497±0.013[3]	−11
Snowy Egret	pre-1943	38	0.241±0.003	
Egretta thula	1970	79	0.220±0.002[3]	−9
Royal Tern	pre-1943	18	0.358±0.004	
Thalasseus maximus	1970	12	0.330±0.007[3]	−8
Olivaceous Cormorant	pre-1943	30	0.347±0.005	
Phalacrocorax olivaceus	1970	24	0.323±0.006[4]	−7
Louisiana Heron	pre-1943	31	0.238±0.003	
Hydranassa tricolor	1970	58	0.225±0.002[3]	−5
Little Blue Heron	pre-1943	31	0.243±0.002	
Florida caerulea	1970	32	0.232±0.003[4]	−5
Great Egret	pre-1943	30	0.295±0.004	
Casmerodius albus	1970	113	0.282±0.002[3]	−4
White Ibis	pre-1943	38	0.363±0.004	
Eudocimus albus	1970	48	0.347±0.003[3]	−4
White-faced Ibis	pre-1943	18	0.312±0.006	
Plegadis chihi	1970	86	0.301±0.002[4]	−4
Black-crowned Night Heron	pre-1943	79	0.278±0.003	
Nycticorax nycticorax	1970	74	0.266±0.003[4]	−4
Black Skimmer	pre-1943	28	0.249±0.004	
Rynchops nigra	1970	48	0.240±0.002[5]	−4
Gull-billed Tern	pre-1943	31	0.239±0.002	
Gelochelidon nilotica	1970	58	0.231±0.002[4]	−3
Laughing Gull	pre-1943	27	0.270±0.003	
Larus atricilla	1970	65	0.263±0.002[5]	−3
Sandwich Tern	pre-1943	25	0.286±0.004	
Sterna sandvicensis	1970	19	0.277±0.005	−3
Anhinga	pre-1943	31	0.328±0.004	
Anhinga anhinga	1970	8	0.318±0.007	−3
Roseate Spoonbill	pre-1943	32	0.426±0.008	
Ajaia ajaja	1970	53	0.415±0.004	−3
Reddish Egret	pre-1943	47	0.270±0.002	
Dichromanassa rufescens	1970	54	0.267±0.003	−1
Least Tern	pre-1943	22	0.156±0.003	
S. albifrons	1970	15	0.154±0.004	−1
Forster's Tern	pre-1943	26	0.219±0.003	
S. forsteri	1970	41	0.218±0.003	0
Caspian Tern	pre-1943	15	0.336±0.005	
S. caspia	1970	32	0.339±0.003	+1

[1] Pre-1947 white and brown pelican data are from Anderson and Hickey (*2*).
[2] All pre-1943 eggs are from the Texas Coast except white pelican, western United States; black-crowned night heron, South Carolina, Florida, and California; snowy egret, little blue heron, great egret, and anhinga, Gulf Coast, Florida, and South Carolina.
[3] $p<0.001$ (Student's t-test).
[4] $p<0.01$.
[5] $p<0.05$.

FIGURE 1. *Location of colonies of wading birds sampled for eggshell thinning, Texas Gulf Coast—1970*

PCB residues were found in all but two species; highest levels occurred in the olivaceous cormorant, Caspian, Forster's, and royal terns (Table 2). Except for the royal tern, these birds feed most frequently in freshwater and estuarine areas. The colonies associated with highest PCB contamination are Vingtun Island near the sprawling urban-industrial complex of Houston-Baytown, Texas, and Dressing Point, south of Freeport, Texas; both areas have numerous oil refineries and petrochemical plants.

Insecticide and PCB residues in marine birds were generally lower and more uniform than levels in birds feeding in freshwater and brackish habitats. ΣDDT and dieldrin residues were higher in eggs from colonies near agricultural areas where insecticides were heavily used. Higher PCBs were consistently associated with urban and industrial areas.

RESIDUE CORRELATIONS WITH EGGSHELL THICKNESS

ΣDDT or DDE was negatively correlated with shell thickness for the great blue heron ($r = -0.66$; $p < 0.01$), white-faced ibis ($r = -0.64$; $p < 0.01$), gull-billed tern ($r = -0.936$; $p < 0.02$), reddish egret ($r = -0.74$; $p < 0.05$), and brown pelican ($r = -0.61$; $p < 0.05$). PCB residues were negatively correlated only for the reddish egret ($r = -0.72$; $p < 0.05$) and the brown pelican ($r = -0.53$; $p < 0.1$); no correlation was found between any of the remaining insecticide residues and eggshell thickness.

Other insecticides and industrial pollutants may affect shell thickness because many pollutants are capable of altering food chain composition, ecosystem energy flow, and ultimately the bioenergetics of individual populations of birds. The many environmental factors and physiological processes that result in eggshell thinning are not well understood. However, the chemical pollutant most frequently identified with shell thinning is DDE. The authors' data support the findings of others who have reported that DDE is the principal agent correlated with eggshell thinning in wild birds (*3, 7, 16, 26*).

SOURCES OF CONTAMINATION

This study indicates that DDE and dieldrin levels detected in egg samples are related to food habits of adult birds. Flickinger and King (*12*) found wet-weight residues of ΣDDT from 0.2 to 1.6 ppm and dieldrin from 0.4 to 2.8 ppm in three species of freshwater fish that are commonly consumed by fish-eating birds. Maximum ΣDDT residues of 9.3 ppm were found in menhaden (*Brevoortia* sp.) and 6.4 ppm in anchovies (*Anchoa* sp.) collected from 1967 through 1969 from rivers, bays, and estuaries in Texas (*9*). Potential effects of these residue levels in food items are evident from results of other studies showing that 3–4 ppm wet-weight DDE in the diet will cause eggshell thinning in certain species of birds (*16, 22, 23, 30*).

DDT was found in the eggs of six species: great egret, white-faced ibis, Sandwich tern, least tern, gull-billed tern, and roseate spoonbill. Low DDT residues, less than 0.8 ppm, were found in all roseate spoonbill eggs. Frequency of contamination in the other five species ranged from 4 percent in the white-faced ibis to 40 percent (two eggs) in the Sandwich tern. The highest DDT residue found was 1.3 ppm in a Sandwich tern egg. Local contamination through the food chain is possible since DDT residues have been found in a pooled sample of 76 sailfin molly (*Poecilia latipinna*) and in crawfish (*Procambarus clarki*), two common foods of aquatic birds in Texas (*12*). Birds migrating to Mexico have been contaminated also since DDT still was widely used there in 1970. DDT residues occurred in all five species that regularly migrate to Mexico: roseate spoonbill, white-faced ibis, snowy egret, Sandwich tern, and least tern.

SIGNIFICANCE OF RESIDUES

DDE—DDE-induced shell thinning has been summarized for numerous birds (*2, 28*). Residues in eggs reported in the present study are comparable to levels found in wild populations that have experienced reproductive failures. Some laboratory studies indicate that harmful effects other than shell thinning are possible. Longcore (*22*) found reduced survival of ducklings (*Anas rubripes*) hatched from

TABLE 2. *Insecticide and PCB residues in eggs of Texas wading birds, 1970*

Species	No.	Mean Residues±SE Wet Weight			Lipid, %
		ΣDDT [1]	Dieldrin	PCBs	
Great Egret	10	23.24±3.61	0.63±0.14 (*10*)	ND	5.6
Caspian Tern	10	15.13±2.25	ND	16.50±4.51 (*10*)	8.5
Laughing Gull	10	10.35±3.90	0.52±0.34 (*5*)	3.00±2.13 (*2*)	10.6
Black Skimmer	5	9.68±3.02	ND	5.40±1.89 (*5*)	11.0
Least Tern	5	6.94±3.52	ND	2.60±0.81 (*4*)	17.2
Louisiana Heron	5	6.50±2.17	0.16±0.12 (*2*)	2.40±0.81 (*4*)	8.5
Olivaceous Cormorant	5	6.22±2.08	0.30 (*1*)	32.00±5.83 (*5*)	4.7
Great Blue Heron	20	5.55±1.05	0.14±0.09 (*3*)	5.54±1.02 (*20*)	5.4
White-faced Ibis	16	5.33±2.92	0.81±0.22 (*12*)	3.00±2.13 (*8*)	6.2
Gull-billed Tern	10	4.89±2.73	0.18±0.15 (*4*)	1.25±0.33 (*6*)	9.3
Royal Tern	5	4.28±0.88	ND	11.60±2.84 (*5*)	12.7
Roseate Spoonbill	10	3.85±0.88	TR (*2*)	2.10±0.28 (*10*)	5.4
Snowy Egret	10	3.26±1.30	1.06±0.67 (*5*)	2.03±1.24 (*7*)	6.2
Brown Pelican	11	3.23±0.20	ND	9.73±1.38 (*10*)	4.8
Reddish Egret	10	2.52±0.60	ND	1.50±0.29 (*10*)	5.9
Black-crowned Night Heron	10	1.76±0.58	TR (*4*)	ND	5.4
Forster's Tern	10	1.74±0.20	0.47 (*1*)	12.50±4.76 (*7*)	9.1
White Pelican	5	1.38±0.30	ND	0.98±0.97 (*5*)	4.7
Little Blue Heron	5	1.20±0.75	0.12±0.05 (*4*)	1.40±0.37 (*5*)	6.5
Sandwich Tern	5	1.12±0.36	0.72 (*1*)	1.40±0.24 (*5*)	15.2
White Ibis	5	0.41±0.12	TR (*5*)	ND	11.0

NOTE: ND=not detected. TR=trace.
Numbers in parentheses represent number of eggs with residues.
[1] ΣDDT residues found in all eggs sampled.

eggs of hens which had consumed food treated with 10 ppm and 30 ppm DDE. Haegele and Hudson (*15*) also reported increased mortality of young and reduced clutch size in ring doves (*Streptopelia risoria*) fed 40 ppm DDE. DDE fed at 10 ppm and 40 ppm to mallards (*Anas platyrhynchos*) reduced hatching of eggs, although survival of hatchlings to 14 days was unaffected (*16*).

Dieldrin—Dieldrin levels found in the present study are lower than those reported in several studies investigating reproductive success and survival of young birds. Fowler et al. (*13*) reported normal hatching success of purple gallinule (*Porphyrula martinica*) and common gallinule (*Gallinula chloropus*) eggs containing average dieldrin residues of 3.8–17.5 ppm. Pheasants (*Phasianus colchicus*), fed varying amounts of dieldrin, showed no effects on fertility, hatching, or survival associated with yolk residues of up to 52 ppm (*4*). Dieldrin residues in whole eggs would normally average about 26 ppm. Chickens fed up to 5 ppm dieldrin showed no effects on clutch size, hatching, or survival of young associated with egg residues of 4–5 ppm (*14*). In contrast, Baxter et al. (*5*) found second-generation effects: fertility and hatching were significantly lower in eggs of hens that received dieldrin through the egg. Dieldrin above 1 ppm in the eggs of golden eagles (*Aquila chrysaetos*) may cause reproductive problems (*28*), and dieldrin residues of 0.54 ppm are lethal to brown pelican embryos (*7*). In view of the great variation in toxicity of dieldrin to different wildlife species, egg residues greater than 1 ppm must be viewed as hazardous.

PCBs—Laboratory experiments indicate that PCB levels found in the present study do not reflect acute exposure of fish-eating birds, but results of reproductive studies are not so conclusive (*17, 26*). One important consideration is the wide range in species sensitivity to PCBs; Heath et al. (*17*) found a fourfold difference in sensitivity between two gallinaceous species. The complex problems associated with the wide range of sensitivity to PCBs and the varying toxicities of different Aroclors were reviewed by Stendell (*27*). These differences emphasize the difficulties in drawing conclusions about the meaning of residues in eggs of fish-eating birds. But at least five species in this study have sufficiently high egg levels of PCBs to warrant additional research: olivaceous cormorant, Caspian tern, Forster's tern, royal tern, and brown pelican.

Mercury—A pooled sample of 30 white-faced ibis eggs contained 0.18 ppm wet-weight mercury, and 10 great blue heron eggs averaged 0.30 ppm. Fimreite (*11*) reported significantly lowered hatching success in pheasant eggs containing 0.5–1.5 ppm mercury, and Borg et al. (*8*) found similar effects at levels of 1.3–2.0 ppm. However, Heinz (*18*) found no significant effects on mallard reproduction associated with egg residue levels of 1.0 ppm. Herring gull (*Larus argentatus*) chicks hatched from each of 24 clutches that contained mercury between 0.5 ppm and 2.0 ppm (*29*). Thus it seems unlikely that mercury residues in white-faced ibis and great blue heron eggs were high enough to affect reproduction adversely.

White-faced ibis eggs were expected to contain high mercury residues because ibis feed in flooded rice fields where mercury-based fungicides were used on seed, but levels were low compared with those found in other studies (*8, 11, 18, 29*). Great blue heron feed in various freshwater and brackish habitats and had slightly but not significantly greater mercury residues than had ibis. This indicates that mercury is found throughout the coastal environment, at least in feeding areas of both species in the Texas rice belt.

THREATENED SPECIES

One objective of the present study was to identify populations possibly threatened by pesticide contamination. On the basis of recent population trends, residue levels, and shell thinning, the authors believe that the brown pelican, white-faced ibis, reddish egret, laughing gull, and Forster's tern warrant immediate attention. Populations of white pelican, olivaceous cormorant, great blue heron, and great egret showed weak or undetermined population trends and should be watched closely in future years. Results of a Texas brown pelican study were recently published (*21*) and ibis data are being prepared for publication.

Acknowledgment

The authors thank Robert E. White, Iwao Okuno, Dennis L. Meeker, and Ronald E. Powers of the Chemical Research and Analytical Section, Denver Wildlife Research Center, for chemical residue analyses. They express appreciation to Lawrence J. Blus, Eric G. Bolen, James O. Keith, Lowell C. McEwen, and Donald H. White for manuscript review.

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Organochlorine Insecticide and Polychlorinated Biphenyl Residues in Woodcock Wings, 1971–72

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ABSTRACT

Pesticide residues in wings of adult woodcock (Philohela minor) *were used to monitor regional differences in a 1970–71 survey of DDT, DDE, TDE, dieldrin, mirex, and PCBs in Maine, New Hampshire, New York, New Jersey, Pennsylvania, North Carolina, South Carolina, Georgia, Lousiana, Michigan, and Wisconsin. In 1971–72, wings were sampled again to compare levels of organochlorine insecticide residues with those of the previous survey and to delineate differences in residue values between adult and immature woodcock. Three additional states, Massachusetts, Minnesota, and Vermont, and one additional organochlorine insecticide, heptachlor epoxide, were included in the second survey.*

Residue levels in the 1971–72 wings showed the same pattern as that observed in 1970–71: organochlorine insecticide residues were highest in wings collected in the southern states and in New Jersey; residues were lowest in samplings taken in the northern and midwestern states. Residues of DDT, TDE, and dieldrin in the 1971–72 wings were slightly lower than those found in 1970–71. DDE, PCB, and mirex residues were significantly lower ($P < 0.05$, $P < 0.01$, and $P < 0.01$, respectively) in 1971–72. Wings of immature woodcock in Louisiana had significantly lower ($P < 0.05$) mirex residues than did adult wings.

Introduction

The woodcock is well suited for monitoring environmental pollutants because it is a migratory upland game bird distributed throughout the eastern United States from the Mississippi River to the Atlantic Ocean and from Michigan to Florida. Personnel from the Fish and Wildlife Service, U.S. Department of the Interior, monitor reproductive success of woodcock by annually inspecting wings submitted by cooperating hunters. Thus wings are in ample quantity for other studies. The same wings can be used to assess quantities of pollutants which the birds have acquired, largely from their food. The woodcock occurs near or at the top of a terrestrial food chain and subsists on animal material, primarily earthworms (*7, 10*). Earthworms concentrate an array of persistent environmental pollutants in their tissues and are important in the diets of a number of avian species (*2, 3, 4, 5, 6*).

Woodcock wings were first monitored for environmental pollutants in 1970–71. Regional differences were clearly demonstrated and baseline measurements were obtained for later comparisons (*8*). An expanded sampling of wings was undertaken in 1971–72 to compare residues with those found in 1970–71, and to determine whether residues in the wings of adult and immature woodcock differed. This paper reports the findings of the 1971–72 survey.

Methods

Wings were collected in 15 states: Connecticut, Georgia, Louisiana, Maine, Massachusetts, Michigan, Minnesota, New Hampshire, New Jersey, New York, North Carolina, Pennsylvania, South Carolina, Vermont, and Wisconsin. These states provided a suitable geographic distribution and offered the best chance for collecting adequate numbers of wings. Because wings from North Carolina, South Carolina, and Georgia were too few to provide a sample from each state, the wings from these states were combined into one tri-state area sample. Wings from adult and immature woodcock from each state and from the tri-state area were sorted into groups of 25. Five of these groups from each state and five from the tri-state area were randomly selected for analysis.

Wings were plucked and the distal joint was removed. The part remaining was ground in a hand grinder and

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homogenized with the group of 25 which made up the complete sample. A 20-g aliquot was taken for analysis.

Organochlorine pesticides and polychlorinated biphenyls (PCBs) were determined at WARF Institute, Inc., Madison, Wisconsin, by the following procedures:

The 20-g aliquot was dried at 40° C for 96–120 hours, and then ground with sodium sulfate and extracted for 8 hours on a Soxhlet extractor with 105 ml of ethyl ether and 250 ml of petroleum ether. The extract was concentrated on a steam bath and diluted to 50 ml with petroleum ether. A 10-ml aliquot of the extract was cleaned and separated into two fractions by elution through a Florisil column with mixtures of ethyl ether and petroleum ether (5+95 and 15+85). An aliquot of the final elution was passed through a standardized silicic acid column as described by Armour and Burke (*1*).

The pesticides and PCBs were determined by electron-capture gas chromatography under the following conditions:

Chromatograph:	Barber-Coleman Model 5360 Pesticide Analyzer
Column:	4-ft × 3-mm glass, packed with 5 percent DC-200 on 80–100-mesh Gas-Chrom Q
Temperatures:	injector 225° C column 205° C detector 245° C
Carrier gas:	purified nitrogen fllowing at 80 ml/minute
Chromatograph:	Barber-Coleman Model 5000
Column:	4-ft × 4-mm glass, packed with 3 percent OV-17 on 100–120-mesh Gas-Chrom Q
Temperatures:	injector 215° C column 200° C detector 250° C
Carrier gas:	purified nitrogen flowing at 80 ml/minute

The sensitivity level of this method was 0.05 ppm organochlorine pesticide and 0.10 ppm PCBs on a lipid basis. Recovery for organochlorine pesticides ranged between 80 and 95 percent, and PCB recoveries ranged between 75 and 85 percent. None of the residue data has been adjusted for rates of recovery.

Results and Discussion

Table 1 shows the ranges and the means as ppm lipid weight for DDT, DDE, TDE, PCBs, dieldrin, mirex, and heptachlor epoxide residues in adult woodcock wings from 12 states and the tri-state area arranged in approximate geographic order from south to north. DDT and its

TABLE 1. *Ranges and geometric means of organochlorine insecticide residues in adult woodcock wings from 15 eastern/midwestern states, 1971–72*

State	Residues, ppm Lipid Weight						
	DDT	DDE	TDE	PCB	Dieldrin	Mirex	Heptachlor Epoxide
Louisiana	1.88–5.45 2.74	5.74–13.09 9.20	0.52–1.40 0.91	1.65–4.10 2.21	1.27–5.56 1.90	4.70–8.49 6.20	0.52–1.13 0.70
Tri-state area	2.35–11.12 5.90	6.99–27.00 18.69	0.64–2.02 1.42	2.63–4.22 3.24	1.22–4.04 1.88	1.66–5.27 3.14	0.21–1.48 0.58
New Jersey	3.27–8.20 4.90	10.11–25.80 16.96	0.76–2.34 1.25	1.97–4.04 2.92	0.27–0.77 0.43	ND–2.12 0.58	ND —
Pennsylvania	0.29–1.37 0.60	2.11–4.71 3.59	ND–0.17 0.03	0.94–2.07 1.39	0.12–2.99 0.30	0.24–0.78 0.48	ND —
Connecticut	1.28–5.61 2.36	3.38–7.12 6.23	0.16–0.65 0.35	1.52–4.38 2.66	0.12–1.12 0.36	ND–0.38 0.50	ND —
New York	0.29–2.87 1.12	4.16–13.07 6.32	0.14–0.26 0.19	1.37–1.84 1.60	0.15–0.21 0.18	0.28–0.96 0.54	ND —
Massachusetts	0.51–5.41 2.16	8.28–22.65 15.63	0.11–0.73 0.33	4.03–9.58 5.84	0.05–0.91 0.15	ND–0.91 0.24	ND —
New Hampshire	0.68–8.47 1.92	5.96–11.56 8.47	ND–0.47 0.31	1.44–1.90 1.69	0.14–0.59 0.27	ND–0.92 0.45	ND —
Vermont	0.25–0.67 1.36	2.63–3.57 3.33	0.07–0.13 0.12	1.54–2.02 1.75	0.08–0.11 0.09	0.24–1.25 0.54	ND —
Maine	0.36–0.94 0.77	3.24–7.20 5.13	0.06–0.25 0.18	0.96–1.26 1.12	0.06–0.12 0.08	0.34–1.44 0.87	ND —
Michigan	0.24–0.68 0.50	2.28–6.96 3.53	0.06–0.32 0.18	1.02–2.21 1.39	0.07–0.11 0.09	0.59–5.01 1.34	ND —
Wisconsin	ND–0.18 0.10	2.60–4.23 3.15	ND —	0.46–1.22 0.77	0.09–0.82 0.18	ND–1.78 0.85	ND —
Minnesota	0.16–0.47 0.30	1.12–3.34 1.74	ND —	ND–0.48 0.08	0.05–0.06 0.05	ND–0.74 0.21	ND —

NOTE: Tri-state area = North Carolina, South Carolina, and Georgia. Wings from three states were combined because not enough were available from any one state.
ND = not detected.

metabolites are distributed in a similar pattern: geometric means of DDT and its metabolites were highest in the tri-state area (DDT, 5.90 ppm; DDE, 18.69 ppm; TDE, 1.42 ppm) and second highest in New Jersey (DDT, 4.90 ppm; DDE, 16.96 ppm; TDE, 1.25 ppm). Differences in contaminant residues levels were determined by one-way analysis of variance with Duncan's multiple range test. Average TDE residues in woodcock wings from the tri-state area and New Jersey were significantly higher ($P<0.01$) for the tri-state area than for all other states except New Jersey, Massachusetts, and Louisiana. The average level of DDE was significantly lower ($P<0.01$) for Minnesota than for all other states.

The average PCB residue in woodcock wings (5.84 ppm) was significantly higher ($P<0.01$) for Massachusetts than for all other states; PCBs in wings were higher ($P<0.01$) for the tri-state area than for all other states except New Jersey, Connecticut, and Louisiana. The average PCB level was significantly lower ($P<0.01$) in Minnesota than in all other states.

Average dieldrin residues in wings from Louisiana and the tri-state area (1.90 ppm and 1.88 ppm, respectively) were significantly higher than those in all other states. Minnesota had the lowest average residues (0.05 ppm).

Heptachlor epoxide residues were found in adult wings in only two areas: Louisiana and the tri-state area. These two areas were included in the fire ant (*Solenopsis saevissima*) eradication program which used heptachlor in the 1950s. Mirex was substituted for heptachlor in the early 1960s. Heptachlor epoxide residues found in adult wings from Louisiana ranged from 0.52 to 1.13 ppm; the geometric mean was 0.70 ppm. Residues in adult wings from the tri-state area ranged from 0.21 to 1.48 ppm; the geometric mean was 0.58 ppm.

Woodcock wings from the two southern areas, Louisiana and the tri-state area, had consistently higher organochlorine residues other than PCBs. PCB residues were highest in Massachusetts and second highest in the southern areas. Wings from Minnesota had the lowest organochlorine residues except for DDT. Wisconsin had the lowest DDT residues; Minnesota had the second lowest.

Eleven of the 13 states, including those in the tri-state area, were sampled in both 1970–71 and 1971–72 (Table 2). Generally, residues were lower in the second sampling period. DDE, mirex, and PCB residues were significantly lower in 1971–72 than in 1970–71 ($P<0.05$, $P<0.01$, and $P<0.01$, respectively).

The relationship of residue levels among states for the two years was tested by a two-way analysis of variance (Table 3). Residues in both sampling periods were consistently highest in the southern states and in New Jersey. Residues were lowest in the northern and midwestern states.

Table 4 shows ranges and geometric means of organochlorine insecticide residues found in immature woodcock wings. Immature wing residues follow the same pattern as residues in adult wings in all but three instances. Mirex residues were higher in immature wings from the tri-state area than in immature wings from Louisiana. Average PCB residues in immature wings were lowest in New Jersey, Louisiana, and the tri-state area; this is the opposite order of residues in adult wings. Heptachlor epoxide residues were found in adult and immature wings from Louisiana and the tri-state area; heptachlor epoxide was also found in two pools of immature woodcock wings from New Jersey.

TABLE 2. *Geometric means of organochlorinated insecticide residues in woodcock wings from eastern/midwestern states, 1970–71 and 1971–72*

Residue	Geom. Mean, ppm Lipid Weight	
	1970–71	1971–72
DDT	1.48	1.26
DDE [1]	8.79	6.82
TDE	1.41	1.42
Dieldrin	0.31	1.31
Mirex [1]	1.54	1.09
PCB [2]	5.58	1.64

NOTE: See Table 3 for list of states sampled.
[1] Significant at $P<0.05$.
[2] Significant at $P<0.01$.

TABLE 3. *Comparison of organochlorine insecticide residues in adult woodcock wings, 1970–71 and 1971–72*

State	Geom. Mean of Residues, ppm Lipid Weight					
	DDE	DDT	TDE	PCB	Dieldrin	Mirex
Maine	4.71d	0.78de	0.19c	2.18c	0.08b	1.04c
New Hampshire	7.58cd	1.61cd	0.25c	3.08bc	0.19b	0.63c
New York	5.92cd	0.77de	0.15c	3.27b	0.19b	1.04c
Pennsylvania	4.07d	0.70de	0.11c	2.51bc	0.17b	0.46c
New Jersey	16.01ab	5.35ab	0.81b	4.16a	0.53ab	0.63c
Tri-state area [1]	28.56a	9.19a	2.27a	5.24a	2.25a	3.11b
Louisiana	10.83bc	2.33bc	0.66b	3.36b	1.98a	10.25a
Michigan	4.65d	0.63e	0.16c	2.43bc	0.15b	1.39c
Wisconsin	5.05d	0.33e	0.51c	2.14c	0.15b	1.08c

NOTE; Values with the same letter are not significantly different.
[1] See Table 1 for explanation.

TABLE 4. *Ranges and geometric means of organochlorine insecticide residues in immature woodcock wings from seven eastern/midwestern states, 1971–72*

State	Residues, ppm Lipid Weight						
	DDT	DDE	TDE	PCB	Dieldrin	Mirex	Heptachlor Epoxide
Maine	0.51–2.28 1.19	2.56–5.28 4.07	0.16–0.41 0.25	0.75–1.07 0.89	0.06–0.83 0.16	ND —	ND —
Michigan	0.46–4.33 0.92	1.90–9.77 3.16	0.12–2.04 0.23	0.95–1.52 1.18	0.07–0.35 0.20	ND —	ND —
New Jersey	3.10–27.04 6.41	9.40–18.01 13.64	0.46–2.84 1.11	1.93–4.28 2.55	0.61–1.07 0.88	ND —	ND–0.42 0.13
Tri-state area [1]	2.89–18.10 6.82	15.29–47.47 26.03	0.64–4.09 1.46	ND–3.93 2.04	0.76–2.70 1.64	1.80–3.98 2.87	0.26–1.23 0.51
Louisiana	1.93–4.01 2.97	7.42–12.53 9.80	0.46–0.95 0.72	1.27–3.68 2.23	1.32–10.20 2.46	1.43–3.72 2.48	0.45–0.96 0.69

NOTE: ND=not detected.
[1] See Table 1 for explanation.

Mirex levels in wings of adult and immature woodcock from Louisiana are clearly different; the residues in wings from adults were significantly higher ($P<0.05$). Mirex residues in adult wings ranged from 4.70 to 8.49 ppm; the geometric mean was 6.20 ppm. In immature wings, mirex residues ranged from 1.43 to 3.72 ppm; the geometric mean was 2.48 ppm. Mirex residue levels from all other states were very low. No significant difference in residue levels were found between adult and immature woodcock in other states, nor among other organochlorine insecticides.

The authors conclude that woodcock wings can be used to help determine the levels and trends of a variety of environmental pollutants in the eastern United States. Periodic assessment of residues in the wings of this species will provide important monitoring information at nominal cost.

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Chlorinated Hydrocarbons and Mercury in Birds of Lake Päijänne, Finland—1972–74[1]

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ABSTRACT

The levels of mercury, PCBs, DDT and its analogs, lindane, and dieldrin were examined in aquatic birds nesting on the shores of Lake Päijänne, the second largest lake in Finland, which is polluted by a wood-processing industry and urban sewages. The primary food of the 10 species examined was fish. In muscle of about 350 individuals, the highest average residues were PCBs; in livers, mercury was the highest. Lindane was found in some individuals; dieldrin appeared in none. The differences among levels in 1972, 1973, and 1974 were not significant. Some regional differences were found, particularly for mercury. Some PCB contamination was observed near the town of Jyväskylä. DDT was distributed evenly. A stronger correlation existed between residues of PCBs and DDT than between residues of any other compounds. In some gulls, males had higher average residues than had females. The DDT:PCB ratio generally corresponded to that of the North Atlantic Ocean, but the difference among species was great. Higher mercury, PCB, and DDT values existed in adults than in juveniles; higher mercury values existed in livers than in muscles. Black-throated divers had highest mercury residues; in herring gulls, PCBs and DDT were highest. The levels generally correspond to those found in other studies.

Introduction

Authors undertook the present study to discover the levels of chlorinated hydrocarbons and mercury in the aquatic birds of Lake Päijänne, Finland. Simultaneously, the methods of chemical analysis and the chemical structures of the compounds were developed. Data on the birds were collected by the University of Jyväskylä as part of a monitoring study of the chlorinated hydrocarbons and mercury in the food webs of Lake Päijänne, in which residues were analyzed in the higher aquatic plants, plankton, bottom fauna, sediment, fishes, and aquatic birds.

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Adults and juvenile birds were analyzed separately. Juveniles were birds of the same summer, ranging in age from a few days to several weeks. Muscle and liver tissues were analyzed separately.

Concentrations of different residues were analyzed according to age, location on the lake, and species. Attention was also paid to the differences between and ratios of residues in liver and muscle, and to the correlations of different residues to muscle:liver ratios, differences of residue load between the sexes, and the ΣDDT:PCB ratios. The significances of the differences were statistically tested.

Sampling and Collection

Lake Päijänne, the second largest lake of Finland (1100 km^2), has been the object of limnological investigations since 1968 (*32*). It receives waste principally from the three origins shown in Figure 1. The sulphite and sulphate pulp mill wastes of Äänekoski come from the north in the upper part of the watercourse, approximately 40 km upstream from Lake Päijänne. Wastes are also discharged into the northern part of the lake from the town of Jyväskylä via Lake Jyväsjärvi (station 1); these effluents contain urban sewages and paper mill wastes. The third source of wastes is in the center of the lake near station 4, which receives effluents from a sulphite pulp mill and two paper mills of Jamsa, as well as a minor amount of domestic waste. At the northern end of the lake, the content of human sewages is greater than in the center which is contaminated almost exclusively by the wood-processing industry. When flowing from the north to the center (station 3), the water becomes cleaner. Water extending from the central part of the lake (station 4) to the southern part (station 6) is quite clean.

The main sampling sites of the study were stations 1, 4, 5, and 6. From stations 2 and 3, a few birds were obtained for supplementary study. Station 1 is polluted by domestic

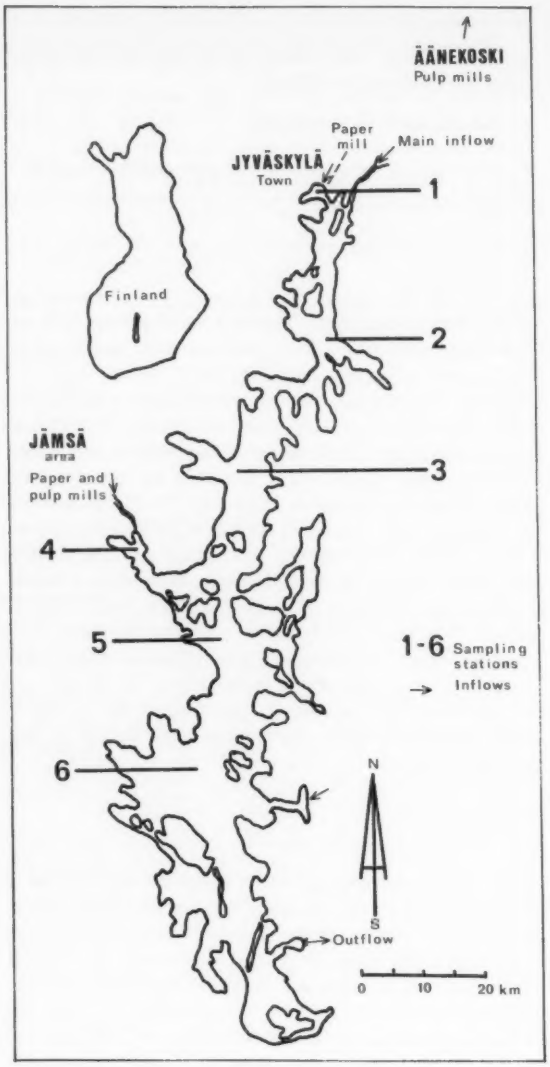

FIGURE 1. *Lake Päijänne with sampling stations.*

sewages and paper mill effluents. Until 1968, effluents from the paper mills and the Äänekoski pulp mills contained mercury originating from slime-preventing chemicals (*12*). Water at stations 2 and 3 gradually becomes cleaner as it moves south. Water at station 4 is affected by a wood-processing industry whose effluents contained mercury until 1968. Water at station 5 becomes cleaner as it approaches station 6, which is almost limnologically pure (*28*).

Adult birds were collected by shooting and young birds were caught live. No individuals were found dead. The adults were all caught after eggs had been laid. The whole birds were conserved by freezing in plastic bags which did not contain PCBs.

Species analyzed were black-throated diver (*Gavia arctica* L.), great crested grebe (*Podiceps cristatus* L.), goldeneye (*Bucephala clangula* L.), redbreasted merganser (*Mergus serrator* L.), sandpiper (*Tringa hypoleucos* L.), lesser black-backed gull (*Larus fuscus* L.), herring gull (*Larus argentatus* L.), common gull (*Larus canus* L.), black-headed gull (*Larus ridibundus* L.), and common tern (*Sterna hirundo* L.).

Species were chosen to represent aquatic birds, especially those which feed at Lake Päijänne in the summer. This is why such species as mallard (*Anas platyrhynchos*) and other common game birds were not sampled. All species, however, are migratory, spending only about one third of the year in Finland.

The number of birds analyzed for total mercury was 344; for methyl mercury, 32; and for chlorinated hydrocarbons, 301.

Analytical Procedures

CHLORINATED HYDROCARBONS

The frozen sample was thawed and 5-10 g breast muscle or liver was weighed. The sample was ground in a mortar with acid-washed sand (Merck) and anhydrous sodium sulphate, 4 g of the latter for each gram of wet tissue. The homogenized mixture was transferred to a glass container and dried at room temperature for 48 hours.

The extraction was performed by Soxhlet in thimbles which had been washed ultrasonically in a 1:1 mixture of acetone and diethyl alcohol. The homogenate was transferred to the thimble and extracted for 6 hours in a mixture of diethyl ether, petroleum ether (boiling point 40°–60° C), n-hexane, and acetone in quantities of 1:9:2, 5:5, 5 (v/v). All solvents were pesticide analytical (p.a.) grade and redistilled. This solvent system has been statistically proved to be the most effective for extracting animal tissue (*14*).

The extracted fat was weighed and cleaned by the following methods: shaking with concentrated sulphuric acid (*2*), thin-layer chromatography (*15*), and a column chromatographic method (*16*). In routine analyses, if extracted fat exceeded 20 mg, it was made into a 1 percent solution in n-hexane and divided into halves. One half was shaken with concentrated sulphuric acid for determining total PCBs, lindane, and DDE. The residues were extracted in hexane which was ready for gas chromatography. The hexane was shaken again with chromic acid for determining DDE (*35*). The second half was applied on a thin-layer plate for determining TDE, DDT, dieldrin, and endrin. When extracted fat was 10 mg or less, thin-layer

chromatography was the only cleanup method used. The column chromatographic method was used mainly for analyzing bird material because residues were greater than in the rest of the samples and required dilution from 10 mg fat, which is the maximum amount accommodated by the column, to 10 ml fat for proper gas chromatography. The cleanup methods have been tested to determine the highest values of the chlorinated hydrocarbons per fresh weight of tissue (*15*). The highest value of PCBs is the only recovery criterion available at present. The sulphuric acid cleanup produced a statisfactory measurement of PCBs in a fat reference sample of the Organization for Economic Cooperation and Development (OECD).

The equipment used in determining the residues was a Varian Model 600 D gas chromatograph with an H^3 electron-capture detector. The length of the glass column was 1.5 m and the inside diameter was 1.5 mm. In the routine analyses the column filling was a mixture of 65 parts of 8 percent QF-1 and 35 parts of 4 percent SF-96 on Chromosorb W 100-120 mesh. Occasionally SF-96 on Chromosorb W 100-120 mesh was also used for control purposes. The carrier gas was nitrogen (99.999 percent). The column temperature was 180° C, the detector and injector were 190° and 225° C, respectively.

The following pesticide standards, all 100 percent pure, were used: aldrin, *p,p'*-TDE, *p,p'*-DDE, *p,p'*-DDT, *o,p'*-DDT, dieldrin, endrin, and lindane. The PCB standard was Clophen A 60 by Bayer because the PCB contamination in Finland had been statistically tested and proved to be that type (*13*). The final concentration in chromatographing was 10 ng/ml for pesticides and 100 ng/ml for the PCBs. The calculation was carried out as described by Gaul (*10*) and the PCBs were calculated by summing nine peaks (total 13 peaks) which did not interfere with the pesticides. Injection of 50 pg pesticides or 500 pg PCBs produced peak heights of approximately 50 percent of full-scale deflection.

TOTAL MERCURY

Total mercury was determined by cold vapor atomic absorption using a Coleman MAS-50 mercury analyzer. A sample of 0.5-1 g was homogenized in an Erlenmeyer flask with 0.5 ml water, and 10 ml concentrated sulphuric acid was added while the flask was kept in an ice bath. The flask was then covered with plastic film and kept in a 60° C water bath for 4 hours. After cooling, 15 ml 6 percent solution of $KMnO_4$ was added from a buret, the bottle was kept in an ice bath and shaken well, and the sample was diluted to 100 ml. To reduce mercury II ions to mercury metal, 2 ml 20 percent hydroxylamine hydrochloride and 1 ml stannous chloride (40 percent solution in 5 percent sulphuric acid) were added and the measurement was taken immediately. The standard was $HgCl_2$ and a standard curve was made daily after treating the standard as described above.

METHYL MERCURY

Methyl mercury was identified by gas chromatography using the following conditions:

Chromatograph: Varian Aerograph 2400
Detector: H^3 tritium
Column: glass, 1.8 m long and 6 mm ID, packed with 10 percent Carbowax 20M on Chromosorb W 80–100 mesh
Temperatures: column: 140° C
injector: 180° C
detector: 210° C

In a Sorvall Omnimixer, 1–5 g material was homogenized in 26 ml 29 percent KBr. Then 3.5 ml 47 percent HBr that had been prewashed with benzene was added to the homogenate which was then centrifuged and the liquid was decanted. The homogenate was treated again with KBr and HBr. The liquid phases were combined in a 250-ml separatory funnel and 50 ml redistilled benzene was added. Methylmercury bromide was added to the benzene. The water phase was extracted again with 25 ml benzene and the extracts were combined; then 8 ml 20 percent cysteine acetate (dried with Na_2SO_4) was added and the solution was shaken to bind the methylmercury bromide to cysteine. Five ml of the water phase was shaken with 1 ml 47 percent HBr and 10 ml benzene to extract the methylmercury bromide in benzene. The benzene phase was chromatographed and the peak heights of the sample and the standard were calculated. Injection of 50 μg Hg as methylmercury bromide produced a peak height of full-scale deflection.

Results

Table 1 shows the average levels of the residues studied in muscles and livers of both adult and juvenile birds. Differences among species, areas, and years are not considered in this table. The table shows that in muscles, the residues of highest concentration are PCBs; in the livers, mercury appears at the highest levels. Concentrations of TDE and DDT are very small compared with those of DDE; all three are combined in subsequent tables as ΣDDT. Lindane was present in only a few individuals, accounting for minute

TABLE 1. *Average chlorinated hydrocarbon and mercury concentrations in muscles and livers of aquatic birds, Lake Päijänne, Finland—1972–74*

Compound	Average Concentration, mg/kg Wet Weight			
	Muscles		Livers	
	Adults	Juveniles	Adults	Juveniles
Total Hg	2.729	0.777	7.900	2.312
Methyl Hg	2.697	0.275	—	—
PCB	4.970	1.135	5.734	0.961
DDE	3.373	0.708	4.187	0.821
TDE	0.012	0.000	0.015	0.000
DDT	0.002	0.000	0.007	0.000
ΣDDT	3.387	0.708	4.209	0.821
Lindane	0.002	0.001	0.000	0.000
Dieldrin	0.000	0.000	0.000	0.000

residue averages. Dieldrin was not present in any individual at concentrations above 0.0005 mg/kg wet weight.

The material of each year of study consists of different numbers of samples from different sampling areas, so results for the different years were not compared with parametric statistical tests. The yearly differences of the average concentrations of total Hg, PCBs, and ΣDDT were examined separately in different bird species for the muscles and livers of the adults and juveniles, using nonparametric Friedman two-way analysis of variance or Wilcoxon matched-pair signed-rank tests (29). No significant differences among the years were observed.

Tables 2–5 present the corresponding residues in birds at different areas of the lake. Because residues vary broadly

TABLE 2. *Average concentrations of total Hg, PCBs, and ΣDDT in muscles of adult birds, Lake Päijänne, Finland*

Species	Statistic	Study area 1			Area 2			Area 3			Area 4			Area 5			Area 6		
		Hg	PCB	ΣDDT	Hg	PCB	ΣDDT	Hg	PCB	ΣDDT	Hg	PCB	ΣDDT	Hg	PCB	ΣDDT	Hg	PCB	ΣDDT
		Residues, mg/kg wet weight																	
Black-throated diver	M	—	—	—	—	—	—	—	—	—	—	—	—	12.80	3.53	5.66	14.57	1.81	6.81
	SD													0.00	2.88	2.59	0.00	0.37	4.34
	N													1	3	3	1	3	3
Great crested grebe	M	2.88	3.99	3.74	—	—	—	—	—	—	1.78	1.33	3.54	—	—	—	—	—	—
	SD	1.13	3.11	2.76							0.80	1.02	3.11						
	N	8	8	8							4	4	4						
Goldeneye	M	—	—	—	—	—	—	—	—	—	0.24	0.22	0.14	—	—	—	—	—	—
	SD										0.06	0.17	0.03						
	N										4	3	3						
Merganser	M	—	—	—	—	—	—	—	—	—	—	—	—	5.48	2.16	1.28	5.42	1.85	3.22
	SD													0.00	0.00	0.00	1.41	1.41	4.36
	N													1	1	1	3	3	3
Sandpiper	M	—	—	—	—	—	—	—	—	—	—	—	—	0.31	0.28	0.71	0.63	3.30	5.41
	SD													0.00	0.00	0.00	0.00	0.00	0.00
	N													1	1	1	1	1	1
Black-backed gull	M	3.00	7.97	1.11	3.44	14.32	5.87	2.43	—	—	2.74	3.64	5.22	3.27	7.15	7.02	3.66	5.10	6.60
	SD	0.00	0.00	0.00	1.24	4.72	1.93	0.60	—	—	1.58	3.09	3.79	1.06	4.91	3.54	1.29	3.29	4.46
	N	1	1	1	5	5	5	3			9	9	9	17	17	17	15	12	12
Herring gull	M	0.10	6.69	1.79	—	—	—	—	—	—	4.00	19.54	20.38	2.80	20.49	6.90	2.97	8.46	6.89
	SD	0.00	0.00	0.00							1.25	2.06	2.36	1.10	12.31	5.10	1.92	5.33	3.92
	N	1	1	1							2	2	2	5	5	5	12	8	8
Common gull	M	2.64	8.98	5.88	2.05	7.16	2.61	3.16	11.77	14.29	2.03	4.04	2.41	2.01	3.33	2.58	1.70	3.18	2.40
	SD	0.00	0.00	0.00	0.15	3.15	2.86	0.00	0.00	0.00	1.15	4.54	2.18	1.48	2.57	2.41	1.01	3.26	1.84
	N	1	1	1	5	5	5	1	1	1	20	20	20	14	14	14	16	16	16
Black-headed gull	M	1.78	4.67	1.58	2.73	4.89	0.87	—	—	—	0.96	2.28	0.69	1.17	2.60	0.48	—	—	—
	SD	0.88	4.17	3.38	1.55	2.53	0.32				0.54	5.84	1.07	0.35	0.14	0.13			
	N	17	16	16	4	4	4				18	18	18	2	2	2			
Common tern	M	3.01	6.18	2.36	2.68	4.53	0.66	5.94	—	—	3.48	2.91	1.37	4.38	2.92	1.32	5.08	4.27	1.53
	SD	1.60	4.38	5.18	2.19	3.03	0.29	0.51			2.28	1.92	1.50	1.32	1.52	0.94	1.61	3.92	1.56
	N	13	12	12	5	5	5	2			16	16	16	7	6	6	7	6	6

NOTE: See Figure 1 for location of study areas.
M = mean, SD = standard deviation, N = number of observations.

TABLE 3. *Average concentrations of total Hg, PCBs, and ΣDDT in muscles of juvenile birds, Lake Päijänne, Finland*

Species	Statistic	Study Area 1			Area 2			Area 4			Area 5			Area 6		
		Hg	PCB	ΣDDT	Hg	PCB	ΣDDT	Hg	PCB	ΣDDT	Hg	PCB	ΣDDT	Hg	PCB	ΣDDT
		Residues, mg/kg wet weight														
Black-throated diver	M	—	—	—	—	—	—	—	—	—	—	—	—	8.15	—	—
	SD													0.00		
	N													1		
Great crested grebe	M	0.53	1.25	0.30	—	—	—	0.38	0.19	0.11	—	—	—	—	—	—
	SD	0.19	0.81	0.15				0.12	0.15	0.04						
	N	6	3	3				3	2	2						
Goldeneye	M	—	—	—	—	—	—	0.07	—	—	0.24	0.15	0.16	—	—	—
	SD							0.00			0.00	0.00	0.00			
	N							1			1	1	1			
Merganser	M	—	—	—	—	—	—	—	—	—	—	—	—	1.32	0.18	0.78
	SD													0.48	0.00	0.00
	N													4	1	1
Black-backed gull	M	1.98	7.70	4.39	0.87	4.06	0.82	—	—	—	0.92	1.10	0.51	0.69	2.28	5.00
	SD	0.00	0.00	0.00	0.00	0.00	0.00				0.23	1.14	0.44	0.41	2.30	5.31
	N	1	1	1	1	1	1				7	3	3	4	3	3
Herring gull	M	0.04	0.67	0.36	—	—	—	0.94	5.44	2.57	0.79	2.97	0.96	0.64	2.85	1.52
	SD	0.00	0.00	0.00				0.00	6.86	3.25	0.00	0.00	0.00	0.41	2.47	0.84
	N	1	1	1				2	2	2	1	1	1	11	3	3
Common gull	M	1.45	1.03	0.40	—	—	—	0.76	0.33	0.13	1.00	0.43	0.51	0.85	1.32	0.59
	SD	0.00	0.00	0.00				0.32	0.00	0.00	0.39	0.51	1.08	0.20	1.90	0.79
	N	1	1	1				3	1	1	13	12	12	6	4	4
Black-headed gull	M	0.45	0.52	0.10	—	—	—	0.35	0.36	0.15	—	—	—	—	—	—
	SD	0.28	0.33	0.05				0.32	0.37	0.11						
	N	9	8	8				11	11	11						
Common tern	M	0.50	0.95	0.16	—	—	—	1.12	1.06	0.67	0.34	1.00	0.41	0.45	0.68	0.25
	SD	0.09	0.22	0.04				0.65	0.59	0.62	0.07	0.35	0.12	0.02	0.00	0.00
	N	5	4	4				4	4	4	3	3	3	2	2	2

NOTE: See Figure 1 for location of study areas.
M = mean, SD = standard deviation, N = number of observations.

TABLE 4. *Average concentrations of total Hg, PCBs, and ΣDDT in livers of adult birds, Lake Päijänne, Finland*

Species	Statistic	Study area 1			Area 2			Area 3			Area 4			Area 5			Area 6		
		Hg	PCB	ΣDDT	Hg	PCB	ΣDDT	Hg	PCB	ΣDDT	Hg	PCB	ΣDDT	Hg	PCB	ΣDDT	Hg	PCB	ΣDDT
		Residues, mg/kg wet weight																	
Black-throated diver	M	—	—	—	—	—	—	—	—	—	—	—	—	45.80	3.79	—	82.33	6.10	24.91
	SD													0.00	0.00	0.00	0.00	0.00	0.00
	N													1	1	1	1	1	1
Great crested grebe	M	5.62	6.37	5.23	—	—	—	—	—	—	6.01	2.82	8.71	—	—	—	—	—	—
	SD	2.19	3.99	4.03							2.90	1.85	7.59						
	N	8	8	8							4	4	4						
Goldeneye	M	—	—	—	—	—	—	—	—	—	2.19	0.30	0.12	2.00	0.35	0.13	—	—	—
	SD										1.32	0.05	0.04	0.00	0.00	0.00			
	N										4	3	3	1	1	1			
Merganser	M	—	—	—	—	—	—	—	—	—	—	—	—	24.00	2.16	1.28	22.97	1.80	3.78
	SD													0.00	0.00	0.00	8.54	0.39	1.93
	N													1	1	1	3	3	3
Sandpiper	M	—	—	—	—	—	—	—	—	—	—	—	—	0.38	0.16	0.44	1.25	0.44	0.25
	SD													0.00	0.00	0.00	0.00	0.00	0.00
	N													1	1	1	1	1	1
Black-backed gull	M	7.20	6.83	3.19	8.74	19.97	16.40	8.42	4.60	4.97	7.98	9.59	9.53	9.74	5.91	8.54	10.62	5.72	7.82
	SD	0.00	0.00	0.00	2.09	16.28	10.99	0.78	2.86	3.55	4.36	8.64	6.96	4.10	5.32	5.22	4.35	3.78	6.54
	N	1	1	1	5	5	5	3	3	3	9	9	9	17	17	17	15	12	12
Herring gull	M	0.21	0.77	2.49	—	—	—	—	—	—	10.65	25.16	19.96	7.61	13.62	6.53	7.59	5.46	4.48
	SD	0.00	0.00	0.00							3.61	6.11	1.23	2.80	9.87	4.96	5.11	2.66	3.62
	N	1	1	1							2	2	2	5	5	5	12	5	5
Common gull	M	6.75	5.33	1.05	6.29	14.16	5.82	10.00	10.07	10.99	5.65	4.45	4.08	6.17	2.48	2.67	5.36	4.58	3.32
	SD	0.00	0.00	0.00	0.72	13.91	2.87	0.00	0.00	0.00	2.74	3.98	4.75	4.58	3.97	3.36	4.45	4.15	3.73
	N	1	1	1	5	5	5	1	1	1	20	19	19	14	14	14	16	16	16
Black-headed gull	M	4.65	6.86	1.11	6.04	4.59	2.34	—	—	—	2.65	2.03	0.66	2.28	2.21	0.65	—	—	—
	SD	2.72	5.17	0.72	3.51	1.94	2.21				1.56	1.74	0.61	0.74	0.28	0.07			
	N	17	17	17	4	4	4				18	18	18	2	2	2			
Common tern	M	7.79	6.19	1.14	6.34	10.35	2.09	15.30	7.10	1.38	8.36	3.87	1.62	15.52	2.62	1.04	14.6	5.35	1.80
	SD	5.06	4.01	0.87	4.68	8.03	1.27	0.28	0.40	0.26	6.32	2.65	1.61	9.52	1.45	0.55	4.32	4.17	1.92
	N	13	13	13	5	5	5	2	2	2	16	15	15	7	7	7	7	7	7

NOTE: See Figure 1 for location of study areas.
M=mean, SD=standard deviation, N=number of observations.

TABLE 5. *Average concentrations of total Hg, PCBs, and ΣDDT in livers of juvenile birds, Lake Päijänne, Finland*

Species	Statistic	Study area 1			Area 2			Area 4			Area 5			Area 6		
		Hg	PCB	ΣDDT	Hg	PCB	ΣDDT	Hg	PCB	ΣDDT	Hg	PCB	ΣDDT	Hg	PCB	ΣDDT
		Residues, mg/kg wet weight														
Black-throated diver	M	—	—	—	—	—	—	—	—	—	—	—	—	19.20	12.62	6.42
	SD													0.00	0.00	0.00
	N													1	1	1
Great crested grebe	M	1.00	1.40	0.29	—	—	—	2.21	0.71	3.55	—	—	—	—	—	—
	SD	0.11	0.34	0.10				0.93	0.48	3.31						
	N	2	2	2				3	3	3						
Goldeneye	M	—	—	—	—	—	—	0.22	0.24	0.13	0.49	0.40	0.30	—	—	—
	SD							0.09	0.05	0.08	0.01	0.17	0.36			
	N							2	2	2	3	3	3			
Merganser	M	—	—	—	—	—	—	—	—	—	—	—	—	2.78	1.81	1.77
	SD													0.63	2.60	2.70
	N													2	3	3
Sandpiper	M	—	—	—	—	—	—	—	—	—	—	—	—	1.11	—	—
	SD													0.04		
	N													2		
Black-backed gull	M	—	—	—	2.30	1.91	0.63	—	—	—	—	—	—	2.10	—	—
	SD				0.00	0.00	0.00							0.00		
	N				1	1	1							1		
Herring gull	M	0.26	0.46	0.14	—	—	—	—	—	—	1.32	0.34	0.18	1.77	—	—
	SD	0.00	0.00	0.00							0.00	0.00	0.00	1.64		
	N	1	1	1							1	1	1	5		
Common gull	M	—	—	—	—	—	—	3.11	1.20	0.42	3.50	0.43	0.72	3.43	0.96	0.37
	SD							1.43	0.00	0.00	1.22	0.48	1.04	1.01	1.33	0.31
	N							4	1	1	8	7	7	4	3	3
Black-headed gull	M	1.45	0.58	0.12	—	—	—	0.56	0.14	0.07	—	—	—	—	—	—
	SD	0.77	0.36	0.09				0.48	0.13	0.07						
	N	4	4	4				6	6	6						
Common tern	M	—	—	—	—	—	—	—	—	—	—	—	—	—	041	0.15
	SD														0.02	0.02
	N														2	2

NOTE: See Figure 1 for location of study areas.
M=mean, SD=standard deviation, N=number of observations.

amont different species, locations, and sampling years, the nonparametric Friedman two-way analysis of variance was used here, too, for comparing the different areas. In areas 1, 4, 5, and 6 (Figure 1) and in the black-backed gull, herring gull, common gull, and common tern, significant regional differences occurred with ΣDDT in the livers of

the adult birds but with no other compounds. The greatest concentration of ΣDDT was in area 4 and the smallest concentration was in area 1.

Table 6 presents averages and standard deviations of total mercury, PCBs, and ΣDDT for the muscles and livers of adults and juveniles. Table 7 presents means and ranges of concentrations in different bird species. From these tables, comparisons between different species, between muscles and livers, and between adults and juveniles can be made. Because lindane was present in only three individuals, the data on this compound appear separately in Table 8.

TABLE 6. *Average Hg, PCB, and ΣDDT concentrations in muscles and livers of aquatic birds, Lake Päijänne, Finland*

Residue		Muscles		Livers	
		Adults	Juveniles	Adults	Juveniles
Total Hg	M	2.73	0.78	7.90	2.31
	SD	1.95	0.86	7.64	2.83
	N	242	101	243	50
PCB	M	4.97	1.14	5.73	0.96
	SD	5.32	1.75	6.33	2.08
	N	229	72	230	40
ΣDDT	M	3.39	0.71	4.21	0.82
	SD	3.94	1.58	5.41	1.68
	N	229	72	230	40

NOTE: M=mean, SD=standard deviation, N=number of observations.

The ratios of residues in muscle to residues in liver were compared with those of other studies (*1, 4, 9, 17, 18*). For mercury, this ratio varied in different bird species between 0.112 and 0.577 in adults, and between 0.278 and 0.573 in juveniles. The muscle:liver ratio for PCBs in adults was between 0.540 and 5.917; in juveniles the ratio was between 0.100 and 8.258. The ΣDDT ratio in adults ranged from 0.383 to 8.884, and in juveniles, from 0.044 to 9.919. These values are approximately the same as those found in the investigations cited above.

Table 9 presents the correlation coefficients of different residues. Compounds whose residues correlated most fre-

TABLE 7. *Concentrations of total Hg, PCBs, and ΣDDT in muscles and livers of aquatic bird species, Lake Päijänne, Finland*

Species	Residue	Muscles				Livers			
		Adults		Juveniles		Adults		Juveniles	
		Mean	Range	Mean	Range	Mean	Range	Mean	Range
		Residues, mg/kg wet weight							
Black-throated diver	total Hg	13.69	12.80–14.57	8.15	8.15	64.07	45.80–82.33	19.20	19.20
	PCB	2.67	1.32– 6.79	—	—	4.94	3.79– 6.10	12.62	12.62
	ΣDDT	6.24	2.90–11.82	—	—	16.27	7.63–24.91	6.42	6.42
Great crested grebe	total Hg	2.51	0.90– 4.76	0.48	0.30– 0.75	5.75	1.15– 8.50	1.73	0.92– 2.80
	PCB	3.10	0.54–10.68	0.83	0.09– 2.18	5.18	0.41–10.00	0.99	0.16– 1.65
	ΣDDT	3.67	0.19– 7.58	0.22	0.08– 0.46	6.39	0.06–17.55	2.25	0.22– 6.89
Goldeneye	total Hg	0.24	0.16– 0.28	0.16	0.07– 0.24	2.15	0.72– 3.42	0.38	0.15– 0.50
	PCB	0.22	0.06– 0.40	0.15	0.15	0.31	0.25– 0.36	0.34	0.21– 0.60
	ΣDDT	0.14	0.13– 0.17	0.16	0.16	0.12	0.09– 0.16	0.23	0.08– 0.71
Merganser	total Hg	5.44	3.80– 6.40	1.32	0.93– 1.97	23.23	16.00–32.50	2.78	2.33– 3.22
	PCB	1.93	0.59– 3.38	0.18	0.18	1.89	1.45– 2.23	1.81	0.30– 4.82
	ΣDDT	2.73	0.56– 8.25	0.08	0.08	3.16	1.28– 5.95	1.77	0.20– 4.89
Sandpiper	total Hg	0.47	0.31– 0.63	—	—	0.82	0.38– 1.25	—	—
	PCB	1.79	0.28– 3.30	—	—	0.30	0.16– 0.44	—	—
	ΣDDT	3.06	0.71– 5.41	—	—	0.34	0.25– 0.44	—	—
Black-backed gull	total Hg	3.25	1.32– 6.56	0.93	0.10– 1.98	9.46	4.50–22.20	2.20	2.10– 2.30
	PCB	6.71	0.27–18.87	2.74	0.38– 7.70	8.00	0.84–47.83	1.91	1.91
	ΣDDT	6.27	0.08–16.83	2.75	0.23–10.97	9.04	1.54–34.41	0.63	0.63
Herring gull	total hg	2.89	0.10– 6.55	0.65	0.04– 1.74	7.53	0.21–18.00	1.49	0.26– 4.62
	PCB	13.49	0.68–37.71	3.30	0.59–19.29	11.27	0.77–29.48	0.40	0.34– 0.46
	ΣDDT	8.26	1.04–22.05	1.60	0.27– 4.87	7.50	0.21–20.83	0.16	0.14– 0.18
Common gull	total Hg	1.97	0.45– 5.36	0.95	0.30– 1.59	5.85	1.22–16.60	3.38	1.42–5.56
	PCB	4.12	0.19–17.45	0.66	0.15– 4.15	4.98	0.33–37.90	0.64	0.09–2.50
	ΣDDT	2.73	0.12–14.29	0.50	0.11– 3.93	3.73	0.02–16.28	0.60	0.09–2.41
Black-headed gull	total Hg	1.48	0.18– 4.36	0.40	0.10– 1.22	3.79	0.64– 9.90	0.92	0.19– 2.52
	PCB	3.51	0.09–24.52	0.43	0.06– 1.34	4.29	0.36–19.13	0.32	0.05– 1.09
	ΣDDT	1.06	0.02–14.13	0.13	0.04– 0.42	1.01	0.09– 5.52	0.09	0.02– 0.21
Common tern	total Hg	3.73	0.69– 8.00	0.64	0.30– 1.92	10.10	0.68–35.60	1.11	1.08– 1.14
	PCB	4.15	0.43–15.92	0.95	3.24– 1.77	5.31	0.76–24.38	0.41	0.40– 0.43
	ΣDDT	1.57	0.16–18.75	0.39	0.12– 1.55	1.47	0.18– 6.55	0.15	0.13– 0.16
Average of all species	total Hg	2.73	0.10–14.57	0.78	0.04– 1.98	7.90	0.21–82.33	2.31	0.15–19.20
	PCB	4.97	0.06–37.71	1.14	0.06–10.29	5.73	0.16–47.83	0.96	0.05–12.62
	ΣDDT	3.39	0.02–22.05	0.71	0.04–10.97	4.21	0.02–34.41	0.82	0.02– 6.89

TABLE 8. *Lindane residues in muscles of three individual birds, Lake Päijänne, Finland*

Species/age	Study Area	Date	mg/kg Wet Weight
Common gull, juvenile	5	1–8–73	0.019
Common gull, juvenile	5	1–8–73	0.058
Merganser, adult (male)	6	5–6–73	0.362

NOTE: See Figure 1 for location of study areas.

TABLE 9. *Correlation coefficients (r) of different residues in muscles and livers of adult and juvenile birds, Lake Päijänne, Finland*

Tissue	Residue	Total	Methyl Hg	PCBs
Muscles Adult	methyl Hg	+0.287***		
	PCBs	+0.214***	+0.049	
	ΣDDT	+0.237***	+0.026	+0.565***
Muscles Juvenile	methyl Hg	+0.039		
	PCBs	+0.114	−0.018	
	ΣDDT	+0.074	−0.012	+0.700***
Livers Adult	PCBs	+0.131*		
	ΣDDT	+0.317***		+0.644***
Livers Juvenile	PCBs	+0.819***		
	ΣDDT	+0.543***		+0.689***

NOTE: * = $p < 5$ percent.
*** = $p < 0.1$ percent.

quently in the greatest number of birds were PCBs and ΣDDT.

Table 10 presents the percentage of total mercury which is methyl mercury. Percentages varied in different species between 91 and 117, indicating inaccuracy of analytical methods, since the correct value must be below 100 percent.

TABLE 10. *Ratios of methyl mercury to total mercury in muscles of adult birds from study areas 1 and 4, Lake Päijänne, Finland—1972*

Species	N[1]	Methyl Hg: Total Hg, %[2]	Ratios, Range
Great crested grebe	6	91	67–110
Black-backed gull	4	117	107–126
Herring gull	1	100	100
Common gull	2	111	107–115
Black-headed gull	7	93	66–127
Common tern	10	107	69–160

NOTE: See Figure 1 for location of study areas.
[1] N=number of individuals sampled.
[2] Percentages over 100 indicate inaccuracy in analytical methods.

Table 11 lists t-test findings which indicate that average concentrations of residues in males and females differed significantly. Muscles and livers in adults of each species were tested. In some gull species significant differences were found, and in all cases the average residue concentration in males was higher than in females.

Table 12 presents the ratio of ΣDDT:PCBs among different bird species for comparison with corresponding values in earlier studies (*4, 5, 23, 27, 34*). Generally, birds which have been nesting in industrial areas contain more PCBs in relation to DDT than do individuals nesting far from such areas (*27*). In Lake Päijänne, these ratios never reached the high levels of 9:10 found in more remote areas of the globe, but the average levels do correspond to those of the North Atlantic (*4*). In many species the ratio ΣDDT:PCBs parallels the values for birds in Greenland (*5*). Great differences exist in the ΣDDT:PCB ratios of the different bird species of Lake Päijänne.

Discussion

The nonparametric tests showed no regional differences of concentration patterns among the birds sampled except for ΣDDT in liver. If the material sampled from the different areas is combined on this ground and t-tests are used to search the yearly differences for every species, only the PCB contents of the black-backed gull seem to have decreased. If the absence of any significant variations between the sampling years is regarded as a basic fact, then the regional differences can be examined for every species from material in which the results of the different years are combined. Such an examination indicates that the different gull species and the common tern contain significantly more mercury at areas 3, 5, and 6 than elsewhere; PCBs appear most often at area 1; and in all species, ΣDDT appears in comparatively even amounts at the different sampling areas. Thus for mercury, the regional maximums are not found in the locations of greatest pollution, areas 1 and 4. The same paradox applies to certain other trophic levels of the lake (*24*). An explanation of this might be that mercury is retained within the sediment at the low-oxygenated areas 1 and 4; it does not pass through the food chain as effectively as it would in sediment which is farther from the sources of pollution.

TABLE 11. *Significant t-test differences between residues in adult male and female birds, Lake Päijänne, Finland*

Species	Content	Tissue	Males			Females			Signif.[1] of Diff.
			M	SD	N	M	SD	N	
Black-backed gull	total Hg	muscle	3.51	1.30	25	2.80	1.05	22	*
	total Hg	liver	10.19	4.24	25	8.19	3.36	22	°
Herring gull	total Hg	liver	10.19	4.41	8	5.76	3.98	12	*
Common gull	total Hg	muscle	2.28	1.13	35	1.46	0.98	22	* *
	total Hg	liver	6.99	3.75	36	3.88	2.46	21	* *
	DDE	liver	4.63	4.24	35	2.24	3.26	21	*
	ΣDDT	liver	4.63	4.24	35	2.24	3.26	21	*

NOTE: M=mean; SD=standard deviation; N=number of observations.
[1] Significances: °=p<10%, *=p<5%, **=p<1%

TABLE 12. *ΣDDT:PCB ratios in muscles and livers of aquatic bird species, Lake Päijänne, Finland*

Species	ΣDDT:PCB Ratio			
	Muscles		Livers	
	Adults	Juveniles	Adults	Juveniles
Black-throated diver	2.339	—	3.292	0.509
Great crested grebe	1.185	0.267	1.233	2.281
Goldeneye	0.658	1.073	0.397	0.688
Merganser	1.418	0.429	1.671	0.976
Sandpiper	1.710	—	1.139	—
Black-backed gull	0.935	1.004	1.130	0.329
Herring gull	0.612	0.479	0.665	0.404
Common gull	0.663	0.763	0.750	0.933
Black-headed gull	0.300	0.301	0.235	0.277
Common tern	0.379	0.409	0.278	0.353
Average	0.681	0.624	0.734	0.854

PCBs seem to enter the watercourse from the town of Jyväskylä but their exact origin is unknown. Otherwise there is little regional difference of PCB and ΣDDT contamination in the waterways around Lake Päijänne, indicating that the residues detected in the birds originate primarily in the wintering regions or along the migration routes, or that they reflect the global levels of contamination. The differences between the average residues in adults and those in juveniles show that bioaccumulation occurs as individuals age (Tables 6, 7). Within each species the contents of mercury, PCBs, and DDT were significantly higher in adults than in juveniles. This was seen in all species that had sufficient material for statistical comparison.

Tables 6 and 7 also show that mercury levels are higher in liver than in muscle, and these differences are significant in all species having sufficient material for statistical comparison according to t-tests. Conversely, PCBs and ΣDDT do not accumulate in the liver more than in pectoral muscles.

Correlations between the different pesticide contaminants (Table 9) do not reveal any causes but they do, to a certain degree, illustrate the possible common origin of the different residues, possible similar bioaccumulation in the food chains, or possible similar behavior in metabolism. The high significant positive correlation between the levels of PCBs and ΣDDT indicates that these fat-soluble contaminants behave similarly.

Authors referred to the published literature to compare residue levels of Lake Päijänne birds with levels in the same species in other countries (*1, 3–7, 9, 17–23, 25–27, 33, 34, 36*). It must be remembered, however, that material from Lake Päijänne did not contain birds that were dead. This excluded from the sample those individuals that may have been fatally poisoned by pesticides.

For the goldeneye, merganser, herring gull, and common tern, mercury levels were lower in Lake Päijänne than in Canada; and for the common gull, mercury content was lower than in Norway. For the grebe, PCB levels were lower in Lake Päijänne than in Great Britain; and for the black-backed gull, levels were lower than in the Faeroe Islands north of Scotland. For the merganser, juvenile herring gull, and common tern, ΣDDT levels were lower in Lake Päijänne than in the United States; and for the black-headed gull, ΣDDT residues were lower than in the Po Delta of northern Italy.

In Lake Päijänne, mercury residues for the black-throated diver were greater than in Aberdeen in eastern Scotland, and Canada; and for mergansers and herring gulls, residues were greater than in Canada. In Lake Päijänne, mercury content was higher for mergansers than elsewhere in Finland; higher for black-backed gulls and herring gulls than in the Faeroes; higher for the herring gulls than at Fife, Scotland; and higher for the black-headed gulls than in Norway and Great Britain. Mercury was present in equal concentrations among mergansers in Lake Päijänne and goosanders in the Baltic Sea.

For the black-backed gull in the Faeroes and the black-headed gull of the Po Delta, PCB concentrations were lower than in Lake Päijänne.

For the black-headed gull and the common tern, ΣDDT concentrations were greater in Lake Päijänne than in the Po Delta. For the herring gulls from the Faeroes and the common tern from the Po Delta, PCB levels were equal to those of Lake Päijänne.

Lindane occurred at about the same concentrations in many individual birds from Lake Päijänne as in those from other locations. Fat of aquatic birds of Greenland averaged 0.40 mg/kg of lindane (*5*); aquatic bird eggs of Ireland averaged 0.045 mg/kg (*8*); cormorants in the United States averaged 0.05 mg/kg in liver and whole bodies (*11*). Black-headed gulls in the Po Delta averaged 0.049 mg/kg in the muscle and 0.495 mg/kg in the liver; for the same individuals, maximum values were 0.110 mg/kg for muscle and 1.87 mg/kg for liver (*34*). Although average concentrations of lindane in Lake Päijänne birds were almost zero, the maximum levels were similar to those in the other countries mentioned.

Dieldrin, which did not appear at all in Lake Päijänne birds, has been reported in aquatic birds elsewhere (*8, 11, 18, 22, 30 34, 37*). Values as high as 0.348 mg/kg have been observed in aquatic birds of Utah, although maximum levels range generally from 0.01 to 0.10 mg/kg (*30*).

Comparison of concentrations in various bird species shows that mercury residues are highest in the diver, merganser, common tern, and common gull. PCB contents are highest in the herring gull and common gull, and ΣDDT is highest in the herring gull, black-backed gull, and diver. Considering ΣDDT concentrations in liver

alone, residues are highest in the diver. Differences among the bird species may depend principally on feeding habits, although duration of life, migration routes, and wintering regions also cause differences. The gulls, especially the black-headed gull and the herring gull, feed on garbage as well as fish, and the black-headed gull also eats terrestrial animals living in arable lands.

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Dieldrin, DDT, PCBs, and Mercury Levels in Freshwater Mullet from the Upper Great Lakes, 1975–76 [1]

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ABSTRACT

Freshwater mullet harvested commercially during various seasons of 1975–76 from the upper Great Lakes were analyzed for organochlorine pesticides, PCBs, and mercury. Species analyzed were Catostomus commersoni, C. catostomus, *and* Moxostoma erythruran. *Whole ground fish, mechanically deboned flesh, head, middle, and tail steaks, and various muscles were analyzed for pesticides and PCBs; only edible flesh was analyzed for mercury. Dieldrin ranged from none detected to 0.23 ppm in deboned and whole ground samples, the DDT range was a trace to 0.30 ppm, and PCBs ranged from 0.06 ppm to 0.79 ppm. Levels were also higher in head sections and in high fat-containing medial muscle and belly flap. Mercury levels ranged from 0.03 ppm to 0.28 ppm in the flesh of mullet from Lake Michigan.*

Introduction

Freshwater mullet from the lakes surrounding Michigan have received little attention as significant sources of human food. In their native form, these fish are frequently considered unattractive to consumers because of their intramuscular bony structure and/or their muddy flavor which is characteristic of fish with their particular eating habits. Estimates indicate, however, that mullet could be harvested from Michigan waters at an annual rate approaching one million kg. Two species of the genus *Catostomus* comprise most of the mullet population in Lakes Huron, Michigan, and Superior. The white mullet *(Catostomus commersoni)* is widespread in Lakes Huron and Michigan, the longnose mullet (*C. catostomus*) predominates in Lake Superior, and the golden redhorse mullet (*Moxostoma erythruran*) is available in commercially harvestable quantities from Lake Huron.

In addition to their muddy flavor, these fish have been unpopular with consumers because of the numerous Y bones throughout the fleshy portion of the fish. Recently, however, mechanical means have been developed for separating meat from bone, yielding a boneless minced flesh product. This minced flesh can be used in various consumer products. However, before commercial products can be developed, it has been necessary to determine the levels of environmental contaminants, their seasonal variation, variation of environmental contaminants within different muscles, and location of the fish in representative species from the three lakes concerned.

Sampling Procedures

Mullet were harvested by commercial anglers from Lakes Huron (Saginaw Bay, Standish, and Au Gres, Michigan), Michigan (Epoufette Bay, Epoufette, Michigan), and Superior (Whitefish Bay, Brimley, Michigan) during different seasons of 1975–76. They were readily available from commercial anglers in Saginaw Bay. The fish were less readily available in the upper Lakes Superior and Michigan, so seasonal variation could not be determined specifically. Fish were ice-packed and transported to the laboratory for processing and analyses, usually arriving the day after the catch. Following heading and gutting, fish to be deboned by machine were split into halves and run through the Bibun deboner (Type SD × 13, 5-mm holes), resulting in a minced flesh product separated from bone, skin, and scales. Whole headed and gutted mullet (35–40 cm long) were coarsely ground three times in a Hobart food cutter fitted with chopper attachment. Other whole dressed mullet were filleted into the ventral, dorsal, medial, and belly flap muscles or sectioned into head, midsection, and tail cross slices. Two mullet, 35–40 cm long, were used for each muscle or section study for each catch date for each lake. Muscles or sections were homogenized separately in an Osterizer blender and all samples were frozen

[1] Michigan Agricultural Experiment Station Journal Article No. 8142. Research supported by Upper Great Lakes Regional Commission Technical Assistance Project No. 10520239.

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and held at −23° C in glass jars before being thawed overnight at 4°–5° C for residue analyses.

Analytical Procedures

PESTICIDES AND POLYCHLORINATED BIPHENYLS (PCBs)

Two samples of each fish variable were extracted separately with hexane–acetone (2:1), partitioned with acetonitrile, and subjected to Florisil-Celite column cleanup according to the method of Yadrick et al. (*7*). Solids were determined by drying 2-g samples under vacuum at 90° C to constant weight; lipid was estimated by evaporating an aliquot of the hexane extract to dryness at 70° C under vacuum.

Gas chromatographic analyses were performed with a Tracor 560 gas-liquid chromatograph (GLC) equipped with a ^{63}Ni electron-capture detector and interfaced with a Digital PDP-8e-Pamila GC data system. Instrument parameters and operating conditions follow.

Column:	1.83-m × 4.0-mm ID Pyrex, packed with 3 percent OV-1 on 80–100-mesh Chromosorb W-HP
Temperatures:	column 190° C injection port 230° C detector 300° C
Carrier gas:	nitrogen flowing at 40 ml/minute

Standards were prepared with 99+ percent pure recrystallized dieldrin, *p,p'*-DDT, and *p,p'*-TDE, and Aroclor 1248 in Nanograde hexane. Quantitations were based on peak area for pesticides; the area of three peaks was used to quantitate the PCBs. Standards were run every morning and after every eight or nine samples. Recoveries with this method of extraction and quantitation were 85±2 percent for PCBs and 92±1 percent for dieldrin and DDT compounds; limits of detection were 0.01 ppm for PCBs and 0.001 ppm for dieldrin and DDT compounds. Data presented in this paper are not corrected for recoveries.

Presence of these residues was confirmed by mass spectrometric analysis on a pool of all extracted samples from each lake. The chromatograph used was a Beckman GC-65 interfaced with a DuPont 21–490 mass spectrometer which in turn was interfaced with a Digital PDP-12-LDP computer. Mass spectra were obtained at an ionizing voltage of 70 eV with a source temperature of 210° C.

MERCURY

Mercury was determined from duplicate edible flesh samples for each catch from each lake as total elemental mercury by using flameless atomic absorption spectrophotometry as described by Gomez and Markakis (*2*). Concentrated sulfuric acid was used to digest the samples as described in their Digestion 1 procedure. Recovery was 95±1 percent, and the limit of detection was 0.005 ppm. Values presented are not corrected for recovery data.

Results

Fat, solids, pesticides, and PCBs in whole ground and mechanically deboned mullet from the upper Great Lakes are presented in Tables 1 and 2. Dieldrin content ranged from none to 0.23 ppm. ΣDDT in white mullet caught in Lake Superior in June ranged from a trace to 0.30 ppm. PCBs varied from 0.06 ppm to 0.79 ppm. All levels are below the tolerances for these environmental contaminants established by the Food and Drug Administration (FDA), U.S. Department of Health, Education, and Welfare, although dieldrin levels in the mullet from Lake Michigan are closest to their tolerance level, 0.3 ppm.

Seasonal variation appears to be minor. As much variation occurred in the levels of contaminants themselves as in the levels as they related to the different catch dates.

The Great Lakes Environmental Contaminant Survey analyzed two freshwater mullet under 16 inches long from Lake Huron in 1974 and four in 1975 (*3*, *4*). Values reported there are similar to those in the current study. An earlier analysis of a freshwater mullet revealed 1.14 ppm DDT (*5*). Thus DDT levels may be decreasing. Similar DDT levels were reported in freshwater mullet from Lakes

TABLE 1. *Fat, solids, pesticides, and PCBs in whole ground freshwater mullet, Upper Great Lakes, 1975–76*

Lake	Type	Date of Catch	Fat, %	Solids, %	Dieldrin		ΣDDT		PCBs as Aroclor 1248	
					Wet Tissue	Fat	Wet Tissue	Fat	Wet Tissue	Fat
					Residues, ppm					
Huron	White	February 75	2.63	23.43	0.03	1.10	0.06	2.03	0.54	10.03
	White	May 75	1.20	21.30	0.10	8.84	0.06	4.26	0.54	43.40
	White	August 75	2.70	24.40	0.09	3.15	0.08	2.89	0.79	29.36
	Redhorse	August 75	7.90	31.70	0.11	1.45	0.08	0.98	0.70	8.86
	White	December 75	2.30	21.30	0.16	3.97	0.30	13.31	0.12	5.30
	White	February 76	2.30	23.90	0.04	1.92	0.08	3.57	0.15	6.28
Michigan	Longnose	June 75	4.20	25.25	0.21	5.01	0.23	3.12	0.62	14.05
	Longnose	August 75	5.55	25.65	0.23	4.31	0.27	4.49	0.71	12.67
	White	June 76	1.15	24.00	0.03	2.77	0.03	2.82	0.16	12.99
Superior	White	June 75	2.05	22.35	—	—	Tr[1]	—	0.06	3.12
	Longnose	December 75	3.95	24.25	0.09	2.33	0.14	3.46	0.26	6.55

[1] Tr = 0.005–0.009 ppm.

Ontario and Erie, although dieldrin levels were less than 0.01 ppm (*1*).

Variation in levels of environmental contaminants from head to tail is summarized in Table 3. The head slices which contained the most fat had the highest levels of environmental contaminants. On a fat basis, however, the distribution was more uniform.

Variation in contamination according to muscle content is shown in Table 4. The high-fat medial muscle and belly flap contained the highest amounts of residues. Because the residues are fat-soluble, trimming would be a feasible method of reducing contaminants if the deboned flesh ever exceeded FDA tolerances. Reinert and Bergman (*6*) also found that these areas had higher levels of contaminants in Coho salmon, but they concluded that trimming would have little benefit because residues in the loin muscles were also high.

Mercury levels in the edible flesh (Table 5) were highest in fish from Lake Michigan. Values reported for fish from Lake Huron are close to those reported by the Great Lakes Environmental Contaminants Survey (*3, 4*).

Acknowledgment

The authors thank Estes Reynolds, Food Science and Human Nutrition Department, Michigan State University, for procuring the fish, and Drs. Dawson, Price and Reynolds for help with fish processing. Appreciation is also expressed to Matthew Zabik, Pesticide Research Center, for mass spectrophotometric analyses.

TABLE 2. *Fat, solids, pesticides, and PCBs in mechanically deboned freshwater mullet, upper Great Lakes, 1975–76*

Lake	Type	Date of Catch	Fat, %	Solids, %	Dieldrin		ΣDDT		PCBs as Aroclor 1248	
					Wet Tissue	Fat	Wet Tissue	Fat	Wet Tissue	Fat
					Residues, ppm					
Huron	White	February 75	2.07	22.37	0.01	0.62	0.03	1.84	0.29	14.13
	White	May 75	1.50	19.83	0.06	4.27	0.06	4.14	0.50	33.39
	White	August 75	1.60	19.75	0.07	4.16	0.08	4.63	0.41	25.01
	Redhorse	August 75	5.50	24.85	0.05	0.96	0.04	0.69	0.18	3.22
	White	December 75	2.75	20.70	0.15	5.22	0.20	6.99	0.70	24.38
	White	February 76	2.95	20.25	0.07	2.47	0.10	3.26	0.17	5.88
Michigan	Longnose	August 75	5.23	23.90	0.13	2.37	0.16	2.93	0.49	9.29
	White	June 76	1.83	19.75	0.03	1.90	0.03	1.87	0.26	14.06
Superior	White	June 75	2.15	18.20	Tr[1]	—	0.01	0.56	0.06	2.93
	Longnose	December 75	3.00	21.15	0.07	2.28	0.12	3.88	0.70	23.32

[1] Tr = 0.005–0.009 ppm.

TABLE 3. *Pesticides and PCBs in sections of freshwater mullet, upper Great Lakes, 1975–76*

Lake	Section	Mean Fat (Range), %	Mean Solids (Range), %	Mean Dieldrin		Mean ΣDDT		Mean PCBs as Aroclor 1248	
				Wet Tissue (Range)	Fat (Range)	Wet Tissue (Range)	Fat (Range)	Wet Tissue (Range)	Fat (Range)
				Residues, ppm					
Huron[4]	Head	5.80 (3.65–9.55)	27.77 (23.65–33.55)	0.16 (0.02–0.68)	3.35 (0.45–13.31)	0.24 (0.03–0.98)	4.84 (1.75–19.25)	0.86 (Tr[2]–1.92)	15.06 (Tr–32.40)
	Middle	3.14 (1.72–7.60)	24.13 (22.65–30.00)	0.06 (0.01–0.21)	2.66 (0.58–9.40)	0.07 (0.02–0.19)	2.91 (0.36–8.09)	0.39 (0.14–1.10)	15.54 (3.02–37.36
	Tail	2.04 (0.65–5.25)	26.21 (23.00–30.00)	0.03 (Tr–0.09)	2.34 (Tr–6.68)	0.04 (Tr–0.13)	2.97 (Tr–6.85)	0.18 (Tr–0.34)	12.94 (Tr–23.29)
Michigan[3]	Head	4.77 (2.25–8.20)	26.22 (23.20–30.10)	0.09 (0.05–0.11)	2.25 (1.33–3.10)	0.10 (0.06–0.17)	2.18 (1.94–2.53)	0.56 (0.49–0.61)	15.09 (8.07–18.78)
	Middle	3.82 (1.15–7.05)	24.30 (21.05–26.70)	0.09 (0.02–0.14)	2.17 (1.62–2.94)	0.12 (0.03–0.17)	2.13 (1.94–2.43)	0.29 (0.23–0.35)	11.61 (8.56–16.90)
	Tail	2.13 (1.24–3.80)	23.07 (21.10–24.70)	0.06 (0.02–0.08)	2.58 (1.58–3.95)	0.06 (0.02–0.08)	2.09 (1.94–2.25)	0.26 (0.10–0.46)	15.55 (7.40–30.58)
Superior[4]	Head	2.65 (2.05–3.25)	23.70 (21.90–25.50)	0.06 (Tr–0.11)	1.68 (Tr–3.36)	0.13 (Tr–0.23)	3.34 (Tr–6.68)	0.24 (0.08–0.39)	7.29 (3.16–11.41)
	Middle	2.10 (1.70–2.50)	24.15 (22.20–26.10)	0.02 (0.00–0.04)	0.84 (0.00–1.68)	0.03 (Tr–0.06)	1.17 (Tr–2.34)	0.15 (0.08)–0.22)	6.66 (4.42–8.89)
	Tail	1.70 (1.20–2.20)	22.48 (20.70–24.25)	0.03 (Tr–0.05)	1.21 (Tr–2.42)	0.06 (0.00–0.11)	2.56 (0.00–5.11)	0.14 (0.07–0.21)	7.80 (5.76–9.84)

[1] Based on six catches from Feburary 1975 to February 1976.
[2] Tr = 0.005–0.009 ppm.
[3] Based on three catches from June 1975 to June 1976.
[4] Based on two catches from June 1975 to December 1975.

TABLE 4. *Pesticides and PCBs in muscles of freshwater mullet, upper Great Lakes, 1975–76*

Lake	Muscle	Mean Fat (Range), %	Mean Solids (Range), %	Mean Dieldrin		Mean ΣDDT		Mean PCBs as Aroclor 1248	
				Wet Tissue (Range)	Fat (Range)	Wet Tissue (Range)	Fat (Range)	Wet Tissue (Range)	Fat (Range)
				Residues, ppm					
Huron[1]	Ventral	0.83	19.94	0.02	3.39	0.07	10.06	0.18	32.20
		(0.55–1.05)	(17.25–22.20)	(Tr[2]–0.05)	(Tr–11.20)	(0.01–0.28)	(1.22–38.69)	(Tr–0.52)	(Tr–120.02)
	Lateral line	5.44	24.26	0.08	2.18	0.10	2.43	0.80	16.93
		(1.50–8.25)	(17.25–29.45)	(0.01–0.18)	(0.36–5.78)	(0.05–0.18)	(0.62–5.39)	(0.19–1.17)	(9.44–23.45)
	Dorsal	1.10	20.11	0.02	2.45	0.06	5.27	0.09	10.83
		(0.50–1.90)	(17.05–21.65)	(Tr–0.06)	(Tr–7.10)	(Tr–0.20)	(0.63–20.47)	(Tr–0.17)	(Tr–16.43)
	Belly flap	3.51	21.55	0.10	3.24	0.13	3.91	0.69	21.03
		(1.15–7.05)	(19.00–26.90)	(0.01–0.24)	(0.70–6.36)	(0.03–0.36)	(1.67–6.29)	(0.24–1.53)	(7.06–41.19)
Michigan[3]	Ventral	1.04	20.59	0.06	6.00	0.06	6.32	0.30	29.20
		(0.60–1.45)	(19.85–21.30)	(0.02–0.10)	(4.04–9.83)	(0.03–0.11)	(3.28–11.11)	(0.09–0.46)	(16.59–40.42)
	Lateral line	8.13	26.70	0.22	3.92	0.28	4.32	1.22	22.10
		(2.49–13.95)	(21.30–31.45)	(0.15–0.28)	(1.57–5.52)	(0.16–0.41)	(1.99–6.00)	(1.13–1.31)	(9.47–40.84)
	Dorsal	1.32	20.53	0.05	4.50	0.22	6.91	0.21	15.12
		(0.52–2.30)	(18.85–21.40)	0.02–0.10)	(1.60–8.51)	(0.16–0.34)	(1.80–13.36)	(0.02–0.34)	(3.44–23.38)
	Belly flap	6.13	23.88	0.26	5.98	0.37	7.77	1.33	24.49
		(1.85–11.90)	(19.45–29.40)	(0.19–0.34)	(2.13–7.92)	(0.13–0.55)	3.62–11.95)	(0.35–2.44)	(10.22–49.20)
Superior[4]	Ventral	1.59	19.68	0.04	1.33	0.07	2.37	0.08	7.85
		(0.87–2.30)	(17.90–21.45)	(Tr–0.08)	(Tr–2.66)	(Tr–0.14)	(Tr–4.74)	(0.06–0.10)	(4.92–10.77)
	Lateral line	8.50	28.95	0.12	1.24	0.37	3.74	0.42	5.71
		(6.80–10.20)	(26.75–31.15)	(0.02–0.22)	(0.35–2.13)	(0.03–0.70)	(0.56–6.91)	(0.27–0.57)	(2.75–8.66)
	Dorsal	1.45	19.73	0.02	1.06	0.04	2.22	0.08	7.32
		(0.85–2.05)	(18.60–20.85)	(Tr–0.04)	(Tr–2.12)	(0.01–0.06)	(1.52–2.87)	(0.08–0.08)	(4.73–9.91)
	Belly flap	3.93	24.13	0.09	1.86	0.13	2.48	0.21	6.92
		(2.00–5.85)	(20.00–28.25)	(0.02–0.15)	(0.71–2.95)	(0.01–0.24)	(0.62–4.34)	(0.13–0.28)	(6.70–7.13)

[1] Based on six catches from February 1975 to February 1976.
[2] Tr = 0.005–0.009 ppm.
[3] Based on three catches from June 1975 to June 1976.
[4] Based on two catches from June 1975 to December 1975.

TABLE 5. *Mecury levels in freshwater mullet, upper Great Lakes, 1975–76*

Lake	Type	Date of Catch	Mercury, ppm
Huron	White	February 75	0.03
	White	May 75	0.06
	White	August 75	0.09
	Redhorse	August 75	0.07
	White	December 75	0.06
	White	February 76	0.05
Michigan	Longnose	June 75	0.21
	Longnose	August 75	0.12
	White	June 76	0.28
Superior	White	June 75	0.10
	White	December 75	0.06

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General

Mirex Incorporation in Estuarine Animals, Sediment, and Water, Mississippi Gulf Coast—1972–74 [1]

Armando A. de la Cruz [2] and Kuang Yang Lue [3]

ABSTRACT

Analysis of mirex residues in estuarine animals, sediments, and waters collected from the Mississippi Gulf Coast in 1972–74 showed the following ranges of concentrations: seston, 200–3000 ppb; molluscs, 36–500 ppb; fish, 0–259 ppb; sediment, 3–5 ppb; and water, 0–0.01 ppb. These data indicate that mirex in aquatic environments is localized in animal tissues and bottom substrate and that only a negligible amount is incorporated in the water.

Introduction

In 1971–74, the authors conducted a series of studies on the toxicity and ecological and physiological effects of mirex on nontarget organisms. The three areas of study included residue monitoring and toxicity, effects of mirex on certain ecological processes of plants and animals, and physiological effects on enzyme systems. The results of these studies are cited in a literature review by Lue (*4*).

The ecological aspect of this project emphasizes the incorporation of mirex in the environment through leaching of the insecticide from decaying fire ant bait in the field (*2, 10*). Mirex residues were recovered from seafood from the Atlantic and Gulf Coastal states (*7*), in terrestrial and aquatic invertebrates from Louisiana (*8*), and in other selected organisms (*11*). During these studies, therefore, the authors routinely collected samples from different habitats (*9*). This paper reports mirex residues detected in samples collected from an estuarine environment on the Mississippi Gulf Coast. The animal samples were collected in the fall of 1972, the sediment samples during summer 1973, and the water samples in 1972 and 1974.

Materials and Procedures

COLLECTION OF SAMPLES

The animals were collected manually from the substrate in St. Louis Bay marsh during low tide. Those from Mississippi Sound were collected by using a shrimp trawl. The specimens were rinsed of mud or debris, blotted dry, wrapped in aluminum foil, and frozen until analysis. Water samples were collected in clean, hexane-rinsed 10-liter jugs by directly filling the jugs a few centimeters beneath the water surface. Water samples for mirex analysis were refrigerated when not immediately processed. Waters intended for seston analysis were promptly filtered through AA millipore filters (0.8-μm porosity) in a millipore vacuum-filtration apparatus. Seston is particulate matter suspended in water including plankton, organic detritus, and inorganic silt. Sediments were collected by an Ekman dredge from St. Louis Bay and by a Petersen dredge from Mississippi Sound. The samples were placed in clean, hexane-rinsed wide-mouth specimen jars and refrigerated until extraction.

EXTRACTION OF SAMPLES

Single or pooled (2–10 specimens) whole-body samples of animals were extracted for residue analysis according to the procedure of Naqvi and de la Cruz (*9*). Only the fleshy tissue of molluscs was extracted. Specimens were rinsed with distilled water to remove salt and briefly dipped in hexane to remove any external insecticide contamination. Samples were ground in nanograde hexane and shaken vigorously, and the decanted solvent was evaporated to dryness. Prior to gas-liquid chromatography, the extracts were cleaned by using activated alumina.

Seston samples were extracted according to the procedure in the *Pesticide Analytical Manual* (*3*) for small samples. The filter paper holding the seston was ground in a tissue grinder with acetonitrile. The filter paper was free of mirex when checked for contamination. The extract was concentrated and reduced to a suitable volume for analysis.

[1] Study supported by Agricultural Research Service, U.S. Department of Agriculture, Cooperative Agreement No. 12-14-100-10935.

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Water was extracted with nanograde hexane in 250-ml separatory funnels; 150 ml samples were shaken vigorously with 50 ml hexane three successive times, 3 minutes each time. The three hexane extracts were combined and evaporated to a volume suitable for gas chromatographic analysis.

Samples of 150 g sediment were extracted with 300 ml hexane-isopropanol mixture (3:1) according to the procedure of Markin et al. (*6*). The extract was filtered through Na_2SO_4 and concentrated to 10 ml.

CHROMATOGRAPHY

Extracts of all samples were analyzed in a Barber-Colman Pesticide Analyzer Model 5360 equipped with an electron-capture detector. A 152.4 mm × 3.2 mm glass column was used. Standard injection techniques were used consistently for all samples. Extract volumes (2 μl) were injected. Information about operating parameters of the analyzer can be obtained from the Physiological Laboratory, Department of Zoology, Mississippi State University, Mississippi State, Mississippi 39762. The concentration of mirex was calculated with the following formula:

$$\text{mirex residue} = Vwd_2/Wvd_1$$

where W = weight of the sample in grams, V = volume of final extract in milliliters, v = volume of extract injected in μl, w = weight of the standard injection in nanograms, d_1 = peak height of standard solution, d_2 = peak height of extract. A second column (1.5 percent SP-250, dimethylchlorosilane-treated and acid-washed) was used to confirm the mirex residues recovered from the field samples.

Results and Discussion

Mirex residues in seston and animals collected from St. Louis Bay and Mississippi Sound are summarized in Tables 1 and 2. Concentrations in seston filtered from Mississippi Sound water (1000–3000 ppb) is one order of magnitude higher than in seston from St. Louis Bay (200–800 ppb). Residues in the animals were all below 1 ppm except in the fiddler crab *Uca* (1.3 ppm). The molluscs, i.e., snails, clams, and mussels, from St. Louis Bay, which are basically filter feeders, had slightly higher levels of mirex (36–500 ppb) than did the other invertebrates from Mississippi Sound (0–133 ppb). In an earlier study, Naqvi and de la Cruz (*9*) found 70–410 ppb mirex in snails and clams collected from a similar estuarine habitat. Residues in the fish ranged from 0 to 259 ppb.

The residue levels of sediments from bay and sound were essentially similar (Table 3) and fairly low (2.8–4.6 ppb). These values are, however, much higher than the residue levels detected in the water samples (0.001–0.010 ppb) from Mississippi Sound, St. Louis Bay, and from the

TABLE 1. *Mirex residues in seston*[1] *and animals*[2] *from St. Louis Bay marsh-estuary, November 1972*

Specimen	Biomass Extracted, G	Residues, ppb[3] Col. I	Col. II
Seston	0.07	817.7	920.6
	0.26	204.1	235.0
	0.19	215.9	199.7
	0.10	408.8	376.9
Rangia cuneata (Clam)	8.10	331.3	247.5
	3.00	490.2	450.0
Modiolus demissus (Ribbed mussel)	4.60	183.8	159.8
	3.00	36.7	71.3
Melampus bidentatus (Snail)	3.80	339.2	265.2
	2.90	471.4	415.2
	0.65	81.8	0.0
	0.45	118.2	0.0
Littorina irrorata (Snail)	0.40	130.9	0.0
	0.80	499.9	31.3
	0.70	75.9	89.3
	0.70	37.9	0.0
	0.60	66.5	35.7
Uca sp. (Fiddler crab)	0.30	1302.0	2661.0
Strongylura marina (Atlantic needlefish)	13.80	50.9	47.4

[1] Seston includes suspended particulate matter consisting of plankton organisms, organic detritus, and inorganic sediment filtered from 300 ml of water with 0.8 μm Millipore acetate filter.
[2] Animals were pooled from 2–10 individuals of about the same size. Biomass represents whole tissue, excluding shells and molluscs.
[3] All analyses were done with two columns to verify the mirex residue.

TABLE 2. *Mirex residues in seston*[1] *and animals*[2] *from Mississippi Sound, September 1972*

Specimen	Biomass Extracted, G	Residues, ppb Col. I	Col. II
Seston	0.01	3038.4	2396.8
	0.03	1507.8	2629.8
	0.03	1001.4	4150.5
	0.23	1172.7	1321.1
	0.01	3260.7	2007.4
	0.02	2291.8	2814.9
	0.01	2677.2	2677.2
	0.01	3243.4	3003.7
Sponge	0.61	133.5	231.0
Luidia clathrata (Starfish)	7.24	28.1	37.0
	8.68	24.0	0.0
Lolliguncula brevis (Squid)	5.40	0.0	0.0
	5.97	13.6	0.0
Palaemonetes sp. (Grass shrimp)	2.77	0.0	0.0
	3.39	0.0	0.0
Callinectes sapidus (Blue crab)	15.84	7.6	106.0
	20.80	3.7	0.0
	18.00	6.4	6.4
Squilla empusa (Mantis shrimp)	1.10	128.0	207.8
	1.30	22.0	0.0
Bairdiella chrysura (Silver perch)	8.30	4.8	0.0
Bagre marinus (Gafftopsail catfish)	6.60	1.1	1.8
Porichthys porisissimus (Atlantic midshipman)	11.70	81.6	97.0
	9.70	15.9	9.6
Etropus crossotus (Fringed flounder)	9.20	7.2	—
Symphurus plagiusa Blackcheek tonguefish)	11.30	12.5	11.7
	12.80	11.0	16.5
Cynoscion arenarius (Sand seatrout)	45.00	0.0	0.0
Strongylura marina (Atlantic needlefish)	19.30	259.1	245.4
	18.00	179.9	132.0

[1] Seston includes suspended particulate matter consisting of plankton organisms organic detritus, and inorganic sediment filtered from 300 ml of water with 0.8 μm Millipore acetate filter.
[2] Animals were single speciments; whole-body tissue was analyzed.
[3] All analyses were done with two columns to verify the mirex residue.

Jordan and Wolf Rivers that empty into the bay (Table 4). Spence and Markin (*10*) found that the highest mirex level in natural water was 0.02 ppb. In a separate study (*5*), the authors found 0.01 ppb residue in samples of water collected from a farm pond. The residue data reported in this paper indicate that mirex in aquatic environments is localized in bottom sediments, animal tissues, and in particulate matter, i.e., seston, suspended in the water, and that only negligible amounts of mirex are incorporated in the water (*1*, *10*).

TABLE 3. *Mirex residues in estuarine sediment, Mississippi Gulf Coast—1973*

Sampling Location	Collection Date	Amount Extracted, g	Residues, ppb [1]	
			Col. I	Col. II
St. Louis Bay [2]	5/29/73	100	2.8	5.0
	6/18/73	100	3.9	5.9
	8/26/73	100	3.5	5.3
Mississippi Sound [3]	7/17/73	100	4.6	2.2
	7/19/73	100	3.5	5.2

[1] All analyses were done with two columns to verify the mirex residue.
[2] Collected by an Ekman dredge from the mouth of Catfish Bayou on the western side of the bay.
[3] Collected by a Petersen dredge about 3 km off the Biloxi–Ocean Spring coastline.

TABLE 4. *Mirex residues in estuarine water, Mississippi Gulf Coast—1972–74*

Sampling Site	Collection Date	Amount Extracted, ml	Residues, ppb [1]	
			Col. I	Col. II
Jourdan River [2]	3/1/74	4,000	0.007	0.000
	6/20/74	4,000	0.005	0.000
			0.009	0.003
			0.004	0.000
Wolf River [2]	5/1/74	4,000	0.001	0.001
	7/31/74	4,000	0.004	0.001
			0.007	0.000
St. Louis Bay	5/1/72	4,000	0.000	0.000
	4/4/72	500	0.000	0.000
	11/15/72	500	0.000	0.000
	5/15/73	4,000	0.030	0.001
	6/12/73	4,000	0.000	0.000
	2/22/74	4,000	0.010	0.003
			0.000	0.001
			0.004	0.000
			0.000	0.000
	3/1/74	4,000	0.004	0.002
	4/6/74	4,000	0.000	0.000
Mississippi Sound	9/23/72	4,000	0.000	0.000
			0.004	0.000
	4/6/74	4,000	0.000	0.000

[1] All analyses were done with two columns to verify the mirex residue.
[2] Samples collected a few kilometers inland from St. Louis Bay.

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APPENDIX

Chemical Names of Compounds Discussed in This Issue

ALDRIN	Hexachlorohexahydro-endo, exo-dimethanonaphthalene 95% and related compounds 5%
AROCLOR 1248	PCB, approximately 48% chlorine
DDD	See TDE.
DDE	Dichlorodiphenyldichloroethylene (degradation product of DDT)
DDT	Dichlorodiphenyltrichloroethane
DIELDRIN	Hexachloroepoxyoctahydro-endo, exo-dimethanonaphthalene 85% and related compounds 15%
ENDRIN	Hexachloroepoxyoctahydro-endo, endo-dimethanonaphthalene
HEPTACHLOR EPOXIDE	1,4,5,6,7,8,8-Heptachloro-2,3-epoxy-3a,4,7,7a-tetrahydro-4,7-methanoindane
LINDANE	Gamma isomer of benzene hexachloride (1,2,3,4,5,6-hexachlorocyclohexane) of 99+% purity
MIREX	Dodecachlorooctahydro-1,3,4-metheno-1H-cyclobuta[cd]pentalene
PCBs (polychlorinated biphenyls)	Mixtures of chlorinated biphenyl compounds having various percentages of chlorine
TDE	Dichlorodiphenyldichloroethane

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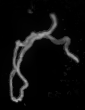
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